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**TO STUDY THE PERMEATION CHARACTERISTICS OF SPICY CHICKEN
FLAVOR USING METALLIZED OPP FILM FROM THAILAND SO AS TO
BE ABLE TO EXTEND THE SHELF LIFE OF BIG JACK (CORN SNACK)**

By

Kanitta Savetpacharaporn

A Thesis

Submitted to the

Department of Packaging Science

College of Applied Science and Technology

in partial fulfillment of the requirements

for the degree of

MASTER OF SCIENCE

Rochester Institute of Technology

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College of Applied Science and Technology

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Rochester, New York

CERTIFICATE OF APPROVAL

M. S. DEGREE THESIS

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has been examined and approved by the thesis committee
as satisfactory for the thesis requirements for the
Master of Science degree

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ROCHESTER INSTITUTE OF TECHNOLOGY
COLLEGE OF APPLIED SCIENCE AND TECHNOLOGY

Title of Thesis: TO STUDY THE PERMEATION CHARACTERISTICS OF
SPICY CHICKEN FLAVOR USING METALLIZED OPP
FILM FROM THAILAND SO AS TO BE ABLE TO EXTEND
THE SHELF LIFE OF BIG JACK (CORN SNACK)

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Abstract

The study of permeation characteristics of flavor as performed to provide the data for the understanding of flavor loss and to prolong the shelf life of the snack food (Big Jack). In this study, six different films were used to compare the permeation characteristics of the Spicy Chicken Flavor and to seek the best packaging material suitable to package the product. The MAS 2000 Organic Permeation Detector was used to collect all the experimental permeation data.

Linalool, the most volatile chemical presenting in the Spicy Chicken Flavor of Big Jack, was used to represent the Spicy Chicken Flavor. Among of six films, the ABX film (an acrylic coated film) seems to yield the most aroma protection while BSR yield the least aroma protection. ABX, HBS, and 60 MAC showed greater flavor barrier than the control film; therefore, these films could be used in order to extend the shelf life of the product. Further studies were suggested to compare the different films in terms of sensory test. Other compounds in the Spicy Chicken flavor could be tested.

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Introduction

The snack food industry is expected to continue its growth into the 2000's. Snack food encompasses a variety of products, including potato crisps, re-constituted potato snacks, cheese snacks, small snack biscuits, pork scratching and nuts. The common packaging materials used are single ply films (principally polypropylene), laminated films, and metallized films. The reels of film, printed in flexo or gravure by flexible packaging converters are often supplied to the snack food manufactures where they are formed into pillow packs, filled, and sealed on vertical form-fill-seal machines. Hence, the qualities of plastic films become more crucial for better protection and extension of the product's shelf life.

"New technology and applications over the past several years has led to a new era in the history of food packaging, marked by the shift from virtually impervious metal cans and glass bottles to semi-permeable flexible packaging structures. Current applications include films, wraps, boil-in-bags, bags-in-boxes, pouches, etc., used for packaging of condiments, candies, dry mixes, cereals, cookies and crackers, to name but just a few. The growing demand for barrier plastic has created a need to develop a better understanding of the transport properties of the polymeric packaging materials to oxygen, moisture, organic vapors and aroma constituents."¹ For oxygen and moisture barrier standard test methods and procedures are ASTM E96-66, ASTM F1249-90 and ASTM D3985-81 for determining transmission rates. But the standard for aroma barrier is hardly found.

¹ Dimitrios Apostolopoulos and Nick winters, *Packaging Technology and Science* 1991 P 131-138

Long range shelf life is the goal of every snack food industry. Three ways to improve the shelf life of the product are:

- A. Improve the product by changing the design of the product or raw material.
For example, if the current snack's shape is too long and prone to breakage, this snack should be redesigned in a shorter length.
- B. Improve the package by redesigning the packaging or by changing the packaging material. For example, the current packaging is made from Low Density Polyethylene (LDPE) which has poor water vapor barrier. If High Density Polyethylene (HDPE) is used instead, the water vapor barrier would be increased.
- C. Change the environment by changing the distribution method. For example, the snack food is packed by filling gas in the bag, and is transported by truck. If the truck passes through a high altitude area, the bag will expand and break the seal. Therefore, by changing transportation by way of train, the product may have a better chance to survive.

Flavor loss is a crucial factor in the shelf life of the product. Aroma barrier relates to the molecular size of the organic vapor, molecular structure of the packaging material, and interaction of the organic vapor and the packaging material. Selection of a good packaging material prevents flavor loss from within the package, and also prevents penetration of odors from outside the package. This will determine the acceptance and success of the snack food, especially when it is flavored. Hence, the results of this study would promote the sales of "Big Jack".

The purpose of this study is to determine the diffusion coefficient (D), solubility coefficient (S), and permeability coefficient (P) of six films of interest. Only the most volatile component in the Spicy Chicken flavor determined from run-

ning Gas Chromatography Mass Spec. was used in this study. All the data were used to evaluate shelf life of the product when packaged in each film.

The Problem and Its Setting

A. The Statement of the Problem

Current shelf life of "Big Jack" (a corn snack brand) is too short for the distribution. Measuring the permeation characteristics of spicy chicken flavor using metallized OPP film from Thailand, and other five films of interest, will provide the data for extending the shelf life of the product.

B. The Subproblems

1. The First Subproblem.

The present shelf life of Big Jack is only one month, which is too short.

2. The Second Subproblem.

The second subproblem is to analyze the present permeation rate of spicy chicken flavor using the metallized OPP film from Thailand.

3. The Third Subproblem.

The third subproblem is to compare the above permeation rate of spicy chicken flavor with other metallized films (ABX, HBS, 60 MAC, MET-HB BSR) from Mobil Chemical Company, USA.

4. The Fourth Subproblem.

The fourth subproblem is to establish if there is a relationship between the spicy chicken flavor permeation characteristics of the film from Thailand and also films from Mobil Chemical Company, USA.

C. Hypothesis

The shelf life of the Big Jack can be further extended by replacing the currently used film with one of the other five commercially available films.

D. Delimitation

1. The study did not include the oxygen barrier characteristic of the polymer films.
2. The study did not include the water vapor barrier characteristic of the polymer films.
3. The effect of light was not included in this study.
4. The study did not include other flavors in the corn snacks, but only focused on the spicy chicken flavor.

E. Definitions of Terms

1. Big Jack

Big Jack is a corn snack which is made of corn starch by an extrusion machine.

2. Permeation²

According to "Activated Diffusion Model," permeation exists in a three-step process. First, the permeant will collide with the film, and adsorb to the surface. Secondly, the permeant will dissolve into the film. Finally, the permeant will diffuse, and then evaporate out of the film. Diffusion occurs from a region of higher concentration of flavor to a region of lower concentration.

² Philip T. Delassus and Gary Strandburg, *Food Packaging Technology*, 1991, pp 64-73.

a. Permeability

The permeability, P , is a measure of overall migration rate of a permeant through a polymer film.

$$P = D \times S$$

where D = the diffusion coefficient, and

S = the solubility coefficient.

b. Diffusion coefficient

The diffusion coefficient is a kinetic term that describes how fast a permeant molecule moves in a polymer host. It is also a measure of how much time is required to reach steady state after an initial challenge. The diffusion coefficient is determined by the size of the permeants, and the size and frequency of fluctuations in the openings between the polymer molecules.

c. Solubility coefficient

The solubility coefficient is a thermodynamic term that describes how many permeant molecules move in a polymer host. It is determined by all the usual parameters of solutions including temperature, chemical activities, and inter molecular interactions plus the state of the polymer relative to its glass transition temperature.

3. Spicy Chicken Flavor

Spicy chicken flavor is a premixed flavor. All ingredients are as shown below.

Oleoresin Paprika	.5%
Garlic oil	10%
Coriander oil	10%
Cayenne oil	5%
Turmeric oil	.3%
Chicken Flavor	20%
Cotton seed oil	54.2%
Total	100%

4. Metallized OPP from Thailand

Metallized OPP film from Thailand is a coextrusion laminated film. It has three layers. The first layer is OPP film, 20 microns thick. The second layer is PE, 25 microns thick. The last layer is metallized OPP film, 25 microns thick. The total thickness of this metallized OPP is 70 microns.

5. Shelf Life ³

Shelf life is defined by the Glossary of Packaging Terms as the length of time that a container, or a material in a container, will remain in a saleable or acceptable condition under specified conditions of storage. Shelf life of packaging must be at least equal to shelf life of the product.

6. Linalool

Linalool,⁴ a fragrant compound that can be isolated from a variety of plants, is 3, 7-dimethyl-1, 6-octadien-3-ol. In this study, Linalool is the most volatile component in spicy chicken flavoring which is determined from Gas Chromatography Mass Spec.

³ Steven W. Gyeszly, *Food Packaging Technology* 1991 P 46-50

⁴ T.W. Graham Solomons, *Organic Chemistry Second edition* 1980, P 942

F. Assumptions

1. Spicy Chicken Flavor would permeate through the polymer films.
2. The permeation rate could be analyzed by the MAS 2000.
3. Other polymer films might offer better flavor barrier properties.

Chapter 1

Background To Research

"Big Jack" Process

Big Jack was made from corn starch and seasoned with spicy chicken flavor. The process of making Big Jack started with the mixing of corn starch with water. Then it is extruded by the extruder machine (TAM, Japan). All products from the extruder machine are called puffed-products. They are then sprayed with oil, and coated with flavor. A puffed product is two inches long, and weighs one gram. The ratio between puffed product and flavor is 1:20 by weight. The final step is baking all the coated puffed products in the long oven for approximately seven minutes at 100°C. Before packing, finished products are dried, and cooled by fans. The finished product is 1.75 inches long and two grams in weight. The vertical form seal machine was used for primary packing before placing them into the shipping container, a B-Flute corrugated box. The box dimensions are 12" X 19" X 23" (see process flow chart on page 50).

Flavor¹

Flavor is a very important characteristic in food and food products. Flavor is often confused with taste. However, while they are related, it must be realized that flavor is the end result, as taste contributes to flavor. The two main ingredients of flavor are taste and odor. The volatile substances responsible for the odor part of flavor have long caused considerable speculation as to the reason these

¹ Frank A. Lee, Ph.D., *Basic Food Chemistry*, 1975, p140.

compounds possess their particular odor qualities. To improve the acceptability of most snack foods, they need to be flavored. Choosing flavors depends on cost, restricted resources, unwanted side effects, and numerous government regulations. Artificial flavors are essentially imitation flavors. Artificial does not necessarily mean synthetic. Mixing pure chemical compounds to obtain imitation flavors is an art and science much surrounded by secrecy. The common snack's flavors in Thailand are paprika, bacon, barbecue, onion and sour cream, salt, tomato, cheese, shrimp, squid, crab, sea food, pizza and caramel. The common snack's flavors in The United States are paprika, bacon, barbecue, onion and sour cream, salt, cheese, mesquite BBQ, ranch and pizza.

Flavor and Volatile²

The volatile substances responsible for the odor of flavor have long caused considerable speculation as to the reason these compounds possess their particular odor qualities. The volatile components generally approach an equilibrium distribution between the food substrates and the head space above it. The amount of volatility depends on vapor pressure of organic compounds in flavor. Rate of equilibrium is related to the concentration difference of the compound in the product relative to its headspace, and the rate of diffusion from the product itself.

² Frank A. Lee, Ph.D., *Basic Food Chemistry*, 1975, p144.

Flavor Storage³

It is important for the buyer to realize that most artificial flavors contain reactive and unstable substances capable of changing drastically during a relatively short period of storage. These changes can result in a loss of flavor, or even more damaging, the development of unpleasant flavors. Buying small quantities and storing flavor stocks at refrigerator temperatures is suggested. The cooler temperatures may, however, result in precipitation of some of the flavors components while freezing may break emulsions, or cause separation of the components in some other way. It is advisable to consult the supplier for recommendation for storage purposes.

When a flavor has been approved by quality assurance, it is a good practice to package several aliquot of the approved batch in bottles, seal the bottles, and store them at refrigerator temperatures. These samples will be withdrawn, as needed, for comparison with future deliveries.

Gas Chromatography Mass Spec⁴

The gas chromatography part of this equipment is effective in separating the volatile components from the mixtures. The individual compounds eluded from the gas chromatographic column are detected electronically and are graphically presented as peaks on a strip chart recorder. These compounds are passed directly to the mass spectrometer to determine their chemical structure. In cases in which two components are eluded at approximately the same retention time, information concerning the structures of two compounds can be obtained by

^{3,4} Frank A. Lee, Ph.D., *Basic Food Chemistry*, 1975, p144.

sampling at the beginning or ascending part of the peak. The carrier gas used in this equipment is helium.

The identification of chemical substances found in natural food materials is preliminary to simulating the natural flavor with mixtures of synthetic compounds. It requires the most sophisticated methods of instrumental analysis, including the use of gas chromatography, high-pressure liquid chromatography, mass spectrometry, and infrared spectrophotometry.

Plastic Films in Snack Packaging⁵

A. Snack Packaging

Packaging has changed from rigid container to flexible packaging because of its properties. The majority of the time, flexible packaging is used for snack's packaging. Snack manufacturers expect their packages to help sell the product, to assist in obtaining the maximum possible shelf life, to give at least some protection against mechanical damage during transport and handling, to provide surfaces on which to print legally required information as well as expiration dates, and to be convenient for the consumer to open and reclose. Five major films that have been used for snack packaging are as follows:

1. Clear oriented polypropylene lamination

Oriented polypropylene lamination (OPP) has been used instead of Cellophane film for approximately the last nineteen years. OPP lamination is suitable for corn and potato chips in clear bags. Later, OPP coextrusions are made either by extruding a heat sealable material onto molten

⁵ James E. Scott, *The Future of Coextruded Films in Snack Packaging*, 1983, pp 387-411.

polypropylene exiting the circular blown films die, or by extruding a heat sealable, or an adhesion promoting material, on both sides of the homopolymer polypropylene base sheet after the machine direction (MD) orientation station but before the cross machine direction (CD) tenter frame operation. This film is used for pretzels and related snack packaging where high puncture resistance is needed.

2. Glassine film and lamination

Cellophane/Glassine lamination films have not been recently used in the market. They have been used for large size bags of potato chips. OPP/Glassine lamination is still used in smaller size bags. For multi-pack operations, a wax coated glassine sheet is still used.

3. Coextruded polyolefin films and lamination

Two-mil coextrusion of high density polyethylene and EVA has replaced the waxed glassine "Twin Pack." In the cereal industry, this film has adopted an easy-open feature by switching the sealant material to EVA blends. On the west coast, snack food packagers like to print on white-HDPE/EVA coextrusions surface for better print quality.

4. Metallized and Foil Lamination

Foil-containing Laminations have high protection. However, the most recent high barrier snack application is being filled with lamination containing metallized PET. Metallized films have a unique combination of high barrier characteristics and foil-like aesthetics. These metallized film properties make this segment of the snack packaging market the most dynamic.

5. Coextruded Film Structures

a. Polyethylene Coextrusion Coatings

The machine can coat by coextrusion onto a base sheet. The initial struc-

tures are for the small clear bag market, and are of the following types:

- (1) OPP/Print/PVdc/(MDPE/Surlyn)
- (2) OPP/Print/(Surlyn/Nylon/Surlyn)

These two types of films indicate that they have comparable product protection, consumer appeal, and machinability characteristics. The only drawbacks are in the stiffness and trackability areas while the sealing attributes are improved.

- (3) OPP/Print/PVdc/(W-LDPE/B-MDPE/Surlyn)

These films are for small bag opaque structures. They should have significantly better barrier and sealing properties compared to the present glassine lamination. Furthermore, they should have a very favorable cost as compared with the present metallized webs.

Because of their inherent softness it is not clear that these types of structures can handle the large bag applications, and because of their relatively low barrier characteristics, they presently do not fit the long shelf life category.

b. Polyethylene Coextruded Film Lamination

The coextruded film lamination was qualified for corn chips. One might have expected the subsequent quick development of an opaque version for large bag potato chip. The need for light barrier, flavor barrier, better machinability, and lower cost have influenced the development of new structures as follows:

- (1) OPP/Print/W-LDPE/(B-HDPE/Tie/EVOH/Tie/Surlyn-EVA)
- (2) OPP/Print/(Surlyn/Nylon/Surlyn)/(W-HDPE/B-HDPE/Surlyn-EVA)
- (3) OPP/Print/Adh./PVdC/(W-HDPE/B-HDPE/Surlyn-EVA)

These films structures have:

- comparable consumer appeal (with better graphics but somewhat less stiffness)
- equal or slightly worse machinability characteristics (essentially tracking).

c. Polypropylene Coextruded Film Lamination

The next logical step for OPP producers is to develop sealable opaque sheets for short-shelf-life potato chips. The structures would be of the following type:

- (1) OPP/Print/W-LDPE/oriented (B-PP/copolymer or PVdC)
- (2) OPP/Print/Adh./oriented(copolymer/(W-PP/B-PP/copolymer or PVdC)
- (3) OPP/Print/W-LDPE/oriented(copolymer/ B-PP/Tie/ EVOH/ Tie/ B-PP/copolymer)

d. Polyolefin Unsupported Webs

The ultimate coextrusion structure is a surface printed oriented structure that has excellent moisture, gas, and flavor barrier; and is sealable over a wide range. The structure would likely be at least a seven-layer structure similar to these barrier sealant films:

- Lacquer/Print/oriented(copolymer/ PP or HDPE/ Tie / EVOH/Tie/ PP or HDPE/ copolymer)

B. Further information for Packaging Requirements for Snack Products

There are four things to consider when choosing snack packaging depending on shelf life. There are three categories: "short" shelf life (< 2 months), "medium" shelf life (2 to 4 months), and "long" shelf life (>4 months).

1. Product Protection

a. Moisture Barrier

Since the majority of salty snack products are essentially dehydrated vegetables, moisture barrier is far and away the most critical product protection characteristic. High temperature and humid weather conditions can turn a crisp potato chip into a soggy, stale product within days. There are two common components which are used in the snack market: OPP and PVdC.

b. Gas Barrier

Normally, the product is surrounded by air within the package. Therefore, there is no necessity for an oxygen barrier. However, if a long shelf life of 4 to 9 months is required, the oxygen-containing atmosphere must be displaced. The most typically treatment is by a nitrogen flushing procedure.

c. Flavor Barrier

The snack producers want their product to taste, as much as possible, like it did emerging from the fryer. While some natural degradation has been found to be acceptable to the consumer, very little outside flavor contamination can be tolerated. But unlike the situation with moisture, companies can have a fair amount of control over odors in their packages' environment. Those firms with a complete store-door distribution system, (comprised of producing plants, OTR trucks, regional warehouses, sales distribution centers, route trucks, and the store display), have the best potential to gain this control.

d. Light Barrier

Most fried snacks are light sensitive, so the packaging should provide light protection. Potato chips held in packages containing either brown pigmented HDPE or foil are stable throughout 10 weeks of storage.

e. Seal Strength

Typical minimum end seal strengths are in the 200 to 400 gm./inch range for packages where the seal is utilized for entry. For those packages exhibiting an alternate entry method (eg., notched end seals), end seal strengths can be beyond 1500 gm./inch.

f. Impact/Puncture Resistance

The values of 2.0 in-lb (Elmendorf test) can adequately prevent potato chip products from puncturing the package. The values of 10 in-lb are required for the sharp tortilla-type products.

g. Tear Resistance

Because there is some propensity for snack products to puncture their package, and since the bag cut-off can cause nicks in the end seals, there is need to specify tear resistance.

h. Durability

The OPP/Glassine lamination is prone to flex cracking that can lead to reduced shelf life.

i. Interlaminar Bond

While there are many who place a great deal of importance on high interlaminar bond values (>150 gm./inch), actual problems have been noted only when a lamination interface has lost essentially all bond strength (<30 gm./inch).

2. Consumer Appeal

a. Graphics

The design must “stand out” from the myriad of other snack designs because the majority of snack food purchases are made on impulse, graphics design, and quality. Many snack packagers are also emphasizing a freshness attribute that they feel. This is obtained by utilizing a foil appearance in the design.

b. Grease Resistance

The finished package must neither be substantially affected by nor be permeable to oils because snack chips are fried in oil. Package integrity must be high enough to prevent oil from leaking out onto the cases and adjacent packages. Flex cracked film and seal leak are the most common causes of oil leakage.

c. Stiffness

The packaging material must be stiff enough to prevent substantial settling of the product, especially in larger supermarket size bags. Moreover, some snack packagers feel that stiff films connote a crispy, fresh product better than softer films.

d. Openability

Packaging should open easily by normal people.

e. Reclosability

The reclosability of snack packages is presently not seen as necessary a feature as it is with related food items. While some of the foil containing structures are more reclosable than others, reclosability appears to have been of secondary importance.

3. Machinability

- a. Trackability
- b. Coefficient of Friction
- c. Sealing Temperatures/range
- d. Hot Tack

4. Costs

- a. Basic film cost
- b. Labor cost

Chapter 2

Review of Related Literature

Flavor/Aroma Effects of Plastic Packaging on Food Product: Setting Quality Guidelines for Measurement of Food and Plastic Packaging Interactions ¹

In the past, plastic film packages were not used for storing the long shelf life food. In the 1960's, homo-polymer and copolymer films and containers were used essentially for short-term (less than one week) storage. These packages could survive by refrigeration. In the 1970's, copolymers and laminated films became more sophisticated, and the packaging applications demanded a longer shelf life of several weeks. During the 1980's, there was an explosion of intricate multilayers to impart barrier characteristics and aesthetics to the package. Extended shelf life has also been demanded, often many months and even years in length. The demands to manufacture high quality resins which perform well in the food packaging industry will continue to be extremely challenging.

The food packaging industry today is actively involved in measuring odor and taste as it relates to maintaining the integrity of food packaging. The flavor ingredients of the food can migrate into the packaging causing the loss of some of its natural aroma profile. Environmental contamination, such as oxygen, water, or other volatile odorants could conceivably migrate through the plastic films to the product. If the appropriate barrier properties are not selected, the food will be adulterated and will develop a bad odor or taste.

¹ Dr. Kent L. Hodges, *PA'91- session C-3*, 1991.

To meet the sanitation packaging, the interaction of food packaging was developed. There are four steps in the process: First, the design and engineering, as well as materials of construction of films, are completed. Then, all the products are tested and measured for barrier properties. These are essentially analytical measurements which determine the permeability, diffusivity, and solubility of migrant molecules through and into the plastic films. The third step, the relationship between aroma and flavor performance measurements, are correlated for the product-packaging interactions. Finally, a food science laboratory measures a function of time for a given packaging application, moisture, texture, and the fate of nutrients.

Innovative BOPP Packaging Films for Flexible Packaging ²

A. In 1985-1986

1. Testing

- a. organoleptic tests (MPE and TNO - Holland) odor protection
- b. gas permeation studies (University of Michigan)
- c. comparing the protective properties of OPP film:
 - (1) coated OPP film
 - (2) non-coated OPP
 - (3) metallized OPP (coated and uncoated OPP)
 - (4) coated paper (PVDC + wax)

2. Conclusion of Testing

- a. Coated BOPP films give superior protection vs uncoated BOPP against most odors.

² L. E. Keller, *Antec'92*, 1992, pp1853-1861.

- b. Coated BOPP films are superior maintaining most flavors vs uncoated - even metallized films.

B. In 1990, Mobil Plastics Europe, and Fraunhofer Institute for Food Technology and Packaging in Munich tested an aroma and flavor protection, light protection, and moisture gain of films when using salt and paprika flavor.

1. Testing

a. aroma and flavor protection

- (1) Sensory tests
- (2) Headspace GC analysis

b. light protection

c. moisture gain

2. Materials

a. Flavors

- (1) Paprika
- (2) Salt

b. Films

- (1) 35 Coex (Printed)
- (2) 35 NB 666 (acrylic coated)
- (3) 50 MW 647 (acrylic coated white opaque)
- (4) 32 MB 777 (Ac/PVdC coated)
- (5) 50 MO 747 (Ac/PVdC coated white opaque)
- (6) 50 MA 757 (Ac/PVdC coated opaque)
- (7) 32 MB 778 (metallized, ctd OPP)
- (8) 20 Coex/met (20 coex laminate)

3. The Conclusions

The test confirmed that:

- a. Moisture affects texture and flavor.
- b. Light affects rancidity, and also causes off-flavor.
- c. Opaque (oppalyte) films provide very good light and moisture protection.
- d. Opaque film MA 757 gives similar protection as metallized films.
- e. Salted chips are more sensitive to the above deterioration than flavored chips

Analysis of Permeation of Food Plastic Monolayers to Methylethylketone (2-Butanone) with The Aromatran ³

A. Two of the most important factors in safe food packaging are to be chemically safe (inert, with no exchange or reaction) and microbiologically safe (it must not contribute to the food product's microbial load or accelerate microbial growth). The storage requirements and mechanical, light and labeling constraints must be taken into account to find the optimum packaging for a given product. One of the most important factors is the packaging material's ability to act as a barrier to atmospheric gases (O_2 , CO_2), and moisture and other solvents or aromas. The methodologies of moisture and oxygen barriers measurements have provided accurate results. However, the accurate measurement, and qualification of aromas and volatile compounds that affect the quality of final product are still required. The Aromatran, a fully automated analytical instrument, is used to testing a methylethyketone at 23°C.

B. Automate Aromatran analyzer is composed of two modules:

- 1. the conditioning module

³ Gilles J. Doyon, Christophe Poulet, Luc Chalifoux, Mathilde Cloutier, Bernard Pascat, Catherine Lorine Lorient and Pierrick Camus, *Packaging Technology and Science*, 1995, pp159-170.

2. the testing (analysis) module

The testing process was controlled by interactive software running on a Philips (P-3464) computer, while test reports were produced on a Roland (DG-P41215) printer. An operational CP-SIL-5CB capillary column (WCOT Fused Silica, Chrompack, NJ, USA), 10 m in length, was used with a flame ionization detector (FID). Experimental conditions were as follows: cold trap in conditioning module at 0°C; pipes at 100°C; cryotrap at -140°C; Subsequent heating to 225°C; oven (column) at 100°C; FID at 225°C.

C. Materials

Plastic film materials were Polypropylene, Polyethylene, and Polyvinyl chloride. All plastic rolls under study were stored vertically, and protected from light. All replicates (five per monolayer or film) were conducted at $23 \pm 0.1^\circ\text{C}$.

D. Gases and vapor (2-butanone) used

The instrument required Hydrogen (H_2), Helium (He), and air (O_2) to operate the testing. Liquid Nitrogen (LN_2) is used for the cryotrap.

E. Statistical analyzes of test results

One-way and two-way Anova variance analyses were carried out on the station state (six measurements) for the five replicates per film at standard deviation of 0.5.

F. Results and Discussion

The experimental calculations yield values of 8,500 and 43,900 $\text{cm}^3 \cdot \mu\text{m}/\text{m}^2 \cdot \text{day} \cdot \text{atm}$ for PP and PE, respectively. This is equivalent to $\pm 1\%$ repeatability. For PVC, a high-transmission material, the value rose to 119,000 $\text{cm}^3 \cdot \mu\text{m}/\text{m}^2 \cdot \text{day} \cdot \text{atm}$, a repeatability of $\pm 2.4\%$. Considering the great variability of PVC, the methodology must be fine tuned.

G. Comments and observations on the system

The Automate Aromatran analyzer is a user-friendly machine but more than 3 months of testing with three materials under study were required to clearly understand end-of-test conditions. The Liquid Nitrogen availability factor (15-30 h for 1801 bottle, and 6-8 h for a 501 bottle) should not be minimized. Since permeated compound is first collected over time in the cryotrap, and then purged through a column, no indication about time is provided. This machine is only able to produce results in terms of quantity. No information about diffusion rates and, subsequently, no information about solubility was possible.

Market Trend and Future Problem in Flavor and Aroma Preserving Films ⁴

The flavor of food is combined of the sense of taste by tongue, flavor by nose, touch on teeth and/or tongue, color and shape of food. Among them, flavor plays an important and delicate role. Only a minute change in the components gives and influences the fancy for food, and determines the value of the goods. The price may jump up by one figure or more by type existence of small amount of flavor as in the case of wine and whisky. It is important to pay full attention to control of the food manufacturing process, the packaging materials, packaging techniques, the storage condition after packed, and the distribution condition.

As flavor and aroma barrier evaluation, there is a sensory test which is adopted as an effective means for food and luxurious food. Recently, instrumental analysis was also carried out. The comparisons of 13 kinds of flavorings and spices is carried out with various packaging material such as EVOH, LDPE, OPP,

⁴ Jingai Cho. *Packaging Japan*, November 1993, pp35-38.

PC and PAN. The results is shown below.

1. PE has little flavor and aroma barrier regardless of its density.
2. PP and PA (poly amide) including BO (bi-oriented) PA have better flavor and aroma barrier than PE which is not sufficient.
3. PET and PC have better flavor and aroma barriers than PP and PA.
4. EVOH, PVA, PAN, and PVDC coated have flavor and aroma barriers of the same or higher level than PET and PC.

A tendency can be seen that a material with better gas barrier has a better flavor aroma barrier.

The Electronic NOSE (Neotronic Olfactory Sensing Equipment) ⁵

The Electronic NOSE identifies odors from solids, gases, and liquids through static head space analysis using an array of conductive polymer sensors integrated with neural network software for pattern recognition. Aroma detection and recognition systems have capabilities of stimulating the human nose. Lab-scale systems were shown. The AromaScan system is based on the interaction of volatile chemicals with an array of semiconductive polymer sensors integrated with neural-network software for pattern recognition. This was developed by AromaScan PLC in U.K., and is made by AromaScan, Inc. (Hollis, NH), USA. Similar systems are Neotronics Olfactory Sensing Equipment (NOSE) from Neotronics Scientific, Inc. (DeMotte, IN). Both can applied on line as well as in lab.

⁵ Charles J. Haberstroh. *Food Engineering*, September 1995, Vol 67, No.9 , pp22.

Chapter 3

Methodology

Introduction

According to Hernandez et al, the recommended method to measure the permeability rates of plastic films to organic vapors is an isostatic method (see schematic diagram in page 52).

The permeation data is obtained by measuring a continuous flow of an organic vapor through a polymer membrane from the initial zero concentration to a steady state condition, as a function of temperature and permeant concentration. The accuracy of the data is very important, hence the instrument for testing must prevent data from variations such as temperature, and concentration changes during the permeation experiment. In this study, the methods, procedures and software used are developed by the MAS Technologies.

Steady State Diffusion Theory¹

The measurement of the flavors permeability values can be extremely slow and very time consuming to reach steady state conditions. The basis theory of the diffusion process is the concentration difference across the medium which was proposed by Fick in 1855. The molecular travel occurs from a high concentration of molecules to low concentration, and that the rate of transfer will be proportional to that concentration difference.

¹ J. A. Ylvisaker, " *Tappi Proceedings, 1995 Polymers, Lamination and Coating Conference Books 2, 1995*" P533-537

Fick's First and second Laws are

$$F = D \, dC/dx \quad (1)$$

$$dC/dt = D \, d^2C/dx^2 \quad (2)$$

where F = the molecular flux in the direction of "x"

C = concentration

D = the diffusion coefficient

In diffusion through a membrane under state condition, Fick's law reduces to the linear equation

$$F = D (C_1 - C_2)/l \quad (3)$$

where C_1 and C_2 = the permeant concentrations on each surface

l = the sheet thickness

Normally, if the concentration of permeant within the film is unknown, the partial pressure values of the opposing gas concentrations will be used. The new equation is

$$F = P (p_1 - p_2)/l \quad (4)$$

where p_1 and p_2 = the test gas partial pressure values on each side of the film

l = the sheet thickness

P = the permeation coefficient

In case of the film concentration is linearly related to partial pressure (a linear isotherm) then Henry's law applies:

$$C = S p \quad (5)$$

where C = the concentration

S = the solubility coefficient

p = the vapor partial pressure

Equation (3) and (4) are equivalent and the relation between the permeability coefficient and the diffusion coefficient is defined by:

$$P = D S \quad (6)$$

where P = the permeation coefficient

D = the diffusion

S = the solubility

The diffusion is the rate of molecular travel and the solubility is the number of molecules percent. The combination of diffusion and solubility describe the total quantity of permeant traversing the film. Since S provides the relationship between the test gas partial pressure and the concentration of the compound within the film, one can use the derived value of S to estimate the tendency of the film material to scalp a compound at any test gas concentration. The measured value of D also provides insight on the rate of concentration changes within the film, insight which is not provided by the Permeation coefficient alone.

Although steady state theory is clear and intuitive, the equations describing molecular flux relative to time are complex. The solution to Fick's law for an isostatic permeation experiment is

$$F(t) = F_e (4\sqrt{\pi}) \sqrt{c} \sum e^{-k c} \quad (7)$$

where F(t) = the flux at time t

$$c = l^2 / 4Dt$$

F_e = the flux at equilibrium

$$k = (2m+1)^2 ; m = 0, 1, 2, \dots \infty$$

at steady state condition.

Another equation is used:

$$R_t = B + R C^{1/2} \sum \{ \exp(-k^2 C) \}$$

for $k = 1, 3, 5, \dots$

where $C = 1 / \{ 4D(t-t_0) \}$

$R = 4P / 1.77245$

R_t = the mass transport rate through a planer surface at time, t

B = the baseline value associated with organic trapped originally in the membrane before test

R = a constant associated with the permeation coefficient

P = the permeation coefficient

C = a constant associated with the diffusion coefficient

D = the diffusion coefficient

t_0 = the instrumental lag time.

Machine & Materials

A. MAS 2000 Organic Permeation Detector

1. Introduction

The MAS 2000 Organic Permeation Detection System is a state-of-the-art laboratory instrument designed to measure the precise level at which organic compound such as flavors, aromas and solvents will permeate, diffuse and solubilize in packaging materials. This highly sophisticated system

incorporates a flame ionization detector, precise temperature and flow control at high speed 486 computer and a very accommodating software package. Although organic permeation has been measured for years by major universities and plastics producers, it is a science which, until today, has not been utilized by general industry (see schematic diagram in page 53).

The main purpose of this study is to evaluate the permeation characteristic of linalool in six different films, namely ABX(Acrylic coated Film), HBS(PVdC coated film), 60 MAC(Acrylic metallized film), MET-HB(Metallized film), Control (Metallized film), and BSR(Coextruded film). A summary of the results can be seen in Table 1 to 7. The full set of permeation curves and result are shown in Figure 1 to 50.

2. MAS 2000 Specifications

System Components:	IBM compatible computer coupled with flow control, temperature control, vertical sample cell and a flame ionization detector.
Instrument Sensitivity:	1 picogram/sec 120 picogram/m ² /sec 1PPB Volumetric
Data Sampling Rate:	10 KHz
Cell Volume:	16 cc
Flow Rate Precision:	±0.1 ml/min
Temperature Range:	ambient to 200° C
Temperature Precision:	±0.05° C
Material Sample Size:	5" x 5" (13 cm x 13 cm)
Sample Measurement Area:	12.6 Sq. inches (.0081 Sq. meters)

Gas Supply Requirements:	pressurized nitrogen, hydrogen & air two-stage regulators (0-100 psi) (0-690 kPa)
Dimensions:	26" D x 36" W x 8" H (66cm x 91cm x 20cm)
Approximate Weight:	80 lbs (30 Kg)
Range of Test Vapors:	all organic gas detectable by flame ionization

B. Gas Chromatography/ Mass Spectrometer

1. The operation of the Gas Chromatography/ Mass Spectrometry (GCMS)

The purpose of using the Gas Chromatography/ Mass Spectrometry is to evaluate the most volatile component present in Spicy Chicken Flavor. As the flavorings are a premix of many other flavorings, it requires sophisticated instrumentation to determine the volatile component. From the test obtained from GCMS, the result showed that the 1,6-Octadien-3-ol, 3,7-dimethyl-,(+) which is, linalool, was present in the highest concentration. (see figures 1 and 2)

2. The operation of the Gas Chromatography

The purpose of using the Gas Chromatography is to confirm and verify the accuracy of the result from the Gas Chromatography/ Mass Spectrometry that was correct. From the test result, the Spicy Chicken Flavor's peak matched with the linalool' peak at retention time 16.1 minutes. (see figures 3-6)

3. Specifications:

Gas Chromatography/ Mass Spectrometer

Machine Type Number: 5995 Hewlett. Packard

Gas Chromatography

Company Name: Hewlett. Packard

Machine Type Number: 5890 series II Plus

C. Chemical

1. Distilled water

Boiling point 100°C

2. Linalool

Chemical Structure: $(\text{CH}_3)_2\text{C}=\text{CHCH}_2\text{CH}_2.\text{C}(\text{CH}_3)(\text{OH})\text{CH}=\text{CH}_2$

Chemical Name: 1,6-Octadien-3-ol, 3,7-dimethyl-(+).

Molecular form: $\text{C}_{10}\text{H}_{18}\text{O}$

Purity: 97 %

FW: 154.25

Boiling point: 194-197 0/720 mm

Lot No: 04027AF

Supplied by: Aldrich Chemical Company Inc., Milwaukee,
Wisconsin 53233, USA.

D. Plastic Films

Six plastic films were selected for the experiment.

1. Metallized film from Thailand (or CONTROL film)

Metallized cast OPP film from Thailand is a coextrusion laminated film. It has three layers. The first layer is OPP film, 20 microns thick. The second layer is PE, 25 microns thick. The last layer is metallized OCPP film, 25 microns thick. The total thickness of this metallized OPP is 70 microns.

2. Procor AB-X or (ABX)

Procor AB-X is a two-side coated, sealable OPP film designed for general use in many applications, including overwrap, horizontal, and vertical packaging. This film can be used unsupported and in lamination. It is suitable for surface and reverse printing. The thickness of film is 0.75 mil.

3. Bicolor 70 HBS-2 or (HBS)

Bicolor 70 HBS-2 is a one-side sealable, one-side high barrier PVdC coated OPP film designed for use as the inside sealant web in lamination. The thickness of film is 0.70 mil.

4. Bicolor 60 MAC or 60 MAC

Bicolor 60 MAC is a one-side acrylic coated, one-side vacuum metallized, high barrier OPP film. This film is designed for adhesive lamination, and is suitable for cold-seal and heat-seal applications. The thickness of film is 0.60 mil.

5. 70 MET-HB or (MET-HB)

Bicolor MET-HB is a vacuum metallized, high barrier OPP film with a proprietary sealant layer. This film offers excellent oxygen and moisture barriers, hot tack, seal integrity, and lap seal range when used within a coextruded outer web. MET-HB is designed specially for adhesive and craze-free extrusion lamination. The thickness of film is 0.70 mil.

6. Bicolor BSR-ONE or (BSR)

Bicolor BSR-ONE is a two-side sealable, one-side treated, coextruded OPP film designed for unsupported plain or surface print

applications on both horizontal and vertical packaging machines. The thickness of film is 0.80 mil.

Experiment Procedure

The Spicy Chicken Flavor was determined by the Gas Chromatography/ Mass Spectrometry to find the most volatile compound. First, the initial time was started at 0.00 minute. Second, the oven was set to the initial temperature of 100°C to the final temperature 250°C. Temperature of oven increased 5°C per minute. After the Temperature Program and Heated Zones were ready, the Spicy Chicken flavor, 0.25 ml, was injected into an injection port. Finally, the Gas Chromatography/ Mass Spectrometry would catch the signals and shown up on the monitor (see figure 1). The signal was interpreted by the computer. The result was 1,6-Octadien-3-ol, 3,7-dimethyl-, (.+ - .) (see figure 2).

The result from the Gas Chromatography/ Mass Spectrometry was confirmed again by using the Gas Chromatography. The operations were started with preparing the Spicy Chicken Flavor in the bottle as well as linalool. The linalool volatile compound was used to register the retention time of linalool which would be compared with the retention time of the Spicy Chicken Flavor. The Linalool headspace was drawn with syringe about 0.25 ml. and injected into the Dynatherm Analytical Instruments, Inc.-Model 890. The volatile compound would evaporate into a thermal tube desorber. The Gas Chromatography would catch the volatile signal after the thermal tube desorber was inserted into the Dynatherm Analytical Instruments. The signal would show on the monitor. In this test, the highest peak at retention time 16.1 minutes was recorded (see figure 3). To confirm the result, the same test would be done again but the volume of the

The result indicates that linalool has a retention time 16.1 minutes, again (see figure 4).

The retention time of linalool was the same as the biggest spike in the Spicy Chicken Flavor. Therefore, linalool is a major component in the Spicy Chicken Flavor. Tests were repeated with 0.25 and 1 ml of Spicy Chicken Flavor's headspace, respectively. The results were in the figures 5 and 6. The test result verified that the result from the Gas Chromatography/Mass Spectrometry was correct. After the Gas Chromatography test was finished, the MAS 2000 Organic Flavor Detector was used to evaluate the permeable characteristics of each film with respect to linalool.

The theory of the MAS 2000 was adapted from an isostatic method, recommended by Professor Ruben Hernandez (see page 55). A specific gas entity was used to exposed one side of a planar sample by means of a constant gas flow within a controlled temperature cell. Compounds which diffused across the sample were swept by a carrier gas to a flame ionization detector. The resulting current signal from the detector was then amplified, and tabulated in real time. The Diffusion and Solubility Coefficients of the sample material for the test permeant were evaluated and determined by regression on the transient solution to Fick's Laws for a planar surface.

Before the test was started, a 6.0"x6.5" film sample was sandwiched between two paperboard film holders with staples (see page 51). The sample was prepared carefully to ensure that the surface of film was perfect and free from dust. The actual diameter of the working area of the film was 12.57 square inches. All the power of machine and computer were turned on including all gas supplies, and was set to a certain pressure. The machine set all variables by using the

using the Test Parameters & Measurement Menu. This menu is composed of File Description, Measurement Parameters, Permeant Parameters and Film Parameters. Now, the test began by choosing the Initiate Measurement from Parameters & Measurement Menu. The sample was inserted between the two cell plates, with the barrier layer facing the back of the unit, towards the detector. Leakage was prevented by isolation of the two chambers from each other by incorporating an elastomeric and compressible Viton O-ring at the front cell. The cell was then closed with an air piston by using the F8 key function. It would assure that the seal area in the vicinity of O ring is free of wrinkles and that sufficient constant pressure is applied to form a leak free seal. The F9 key was used to ignite the detector. After a steady baseline was established on the screen, a calibration mode was entered by using F5 key. The computer would save the current baseline signal as zero value for reference. The calibration, at the first test of each day, was set by injecting .25 cc of the 97 % linalool's headspace into the injection port by an air-tight syringe. From the Ideal Gas Law, the computer automatically compared the resulting integration of that response or spike with the number of molecules of permeant in the syringe injection. The Ideal Gas Law states that the pressure of the gas is inversely proportional to its volume and directly proportional to its concentration and the temperature. $n = PV/RT$ where P is the partial pressure of the injection gas, V is the injection volume, R is the gas constant, T is the temperature in degree Kelvin, n is the injection moles.

From the Ideal Gas Law and the set up parameters, the system calculated the number of moles injected and equates that to the response (integration of spike) to derive a calibration factor. Using this factor later when the signal went up due to permeation, the system could calculate how many moles came through

the film. The calibration factor was calculated from

$$F_C = n/I$$

where n is the injection moles and I is the signal integration value in ampere seconds. The calibration factor will be in the units moles/ampere/second. Then a second calibration factor which associates the detector signal with permeant mass from: $W_C = F_C m$ where F_C is the calibration factor in moles/ampere/second and m is the molecular weight of the permeant in Kg. The factor W_C was calculated in the units Kg/ampere/second.

After the signal had dropped back to original steady baseline, the system would automatically exit the calibration mode and the actual test could begin. The cell temperature was kept constant ($\pm 0.05^\circ \text{C}$) by two electric heaters and a constant flow of Nitrogen gas was flowing continuously through the front cell (or upstream side) and back cell (or down stream side). During the test mode, the Nitrogen stream in the front cell was replaced with the permeant stream. Simultaneously, a constant flow of Nitrogen was also passing through the back cell (downstream side), sweeping any organic permeant molecules from the back cell at a constant rate and conveying them to the detector. The detection system comprised of an Flame Ionization Detector (FID), interfaced to the back cell. At preselected time intervals (every 5 seconds), the amount permeated was tracked and fit to a diffusion curve using the Mass Transport Theory. The amount permeated and diffusion rate were measured, and then the solubility was extrapolated from the equation ($S = P/D$). The transmission rates were monitored continually until steady state conditions were obtained. The computer compared the resulting response with the data obtained during calibration simultaneously and the permeation results were plotted on the screen. The computer received the signal,

which was detected by the FID in terms of pico-amps, and converted it in terms of micro-grams/m². hr. from the calibration factor. A constant concentration of the vapor of linalool was obtained using a midget bubble to bubble Nitrogen gas through the liquid permeant. Function F3 key was used when the testing had been completed. The experiment used three samples of each material for calculation Arrhenius linear graph. This graph was a graph of $1/k$ (k = temperature in degree Kelvin) versus $\ln P$ (P = permeability), expected to be linear. The MAS system was used to project permeation values at different temperatures. Hence, different temperatures were tested for each film sample.

Chapter 4

Results & Discussion

Analysis of Data

ABX Film (see Table 1): As the temperature was increased from 70 °C to 90 °C, the permeation rate of linalool increased by approximately 14.47 times (from 182 to 2,635 ugms.mil/m².hr). The diffusion rate increased by approximately 6.9 times (from 0.08 to 0.552 1/hr). The solubility rate increased by approximately 2.09 times (from 2,275 to 4,773.55 ugms/m²). The permeation rate, the diffusion rate, and the solubility rate of linalool increased when the temperature increased. (see Figure 10: Permeation coefficient, Diffusion coefficient, and Solubility coefficient of linalool versus Temperature.)

HBS Film (see Table 2): As the temperature was increased from 70 °C to 90 °C, the permeation rate of linalool increased by approximately 6.16 times (from 189 to 1,165 ugms.mil/m².hr). The diffusion rate increased by approximately 5.63 times (from 0.186 to 1.049 1/hr). Furthermore, the solubility rate increased by approximately 1.09 times (from 1,016.12 to 1,177.82 ugms/m²). The HBS film has a result data in the same way to the ABX film. The permeation rate, the diffusion rate, and the solubility rate of linalool increased when the temperature increased. Compared to ABX, the HBS film allowed linalool to permeate through easier. (see Figure 14: Permeation coefficient, Diffusion coefficient, and Solubility coefficient of linalool versus Temperature.)

60 MAC Film (see Table 3): As the temperature was increased from 70 °C to 90 °C, the permeation rate of linalool increased by approximately 4.46 times (from 608 to 2,713 ugms.mil/m².hr). The diffusion rate increased by approximately 5.59 times (from 0.114 to 0.638 1/hr). However, the solubility rate decreased by approximately 0.79 times (from 5,333.33 to 4,252.3511 ugms/m²). The permeation rate and the diffusion rate of linalool increased. Conversely, the solubility rate decreased when the temperature increased. The data suggested that as temperature increases, the flavor permeates through the film rather than solubilize within the film. Hence, scalping would decrease. (see Figure 18: Permeation coefficient, Diffusion coefficient, and Solubility coefficient of linalool versus Temperature.)

MET-HB Film (see Table 4): As the temperature was increased from 70 °C to 90 °C, the permeation rate of linalool was increased by approximately 1.41 times (from 373 to 527 ugms.mil/m².hr). The diffusion rate increased by approximately 3.641 times (from 0.592 to 2.156 1/hr). However, the solubility rate decreased by approximately 0.387 times (from 630.068 to 244.43 ugms/m²). The MET-HB has the same result data the 60 MAC. The permeation rate and the diffusion rate of linalool increased, but the solubility rate decreased when the temperature increased. This means that when the temperature is higher, the flavor will permeate through the film more than scalp inside the film. However, the MET-HB seems to have similar permeation behavior to the 60 MAC. (see Figure 22: Permeation coefficient, Diffusion coefficient, and Solubility coefficient of linalool versus Temperature.)

CONTROL Film (see Table 5): As the temperature was increased from 70 °C to 90 °C, the permeation rate of linalool increased by approximately 2.17 times (from 2,150 to 4,686 ugms.mil/m².hr). The diffusion rate increased by approximately 2.6 times (from 0.182 to 0.476 1/hr). However, the solubility rate decreased by approximately 0.46 times (from 30,012.47 to 14,093.75 ugms/m²). The permeation and diffusion rate increased when the temperature increased, but the solubility rate decreased. Once again, the scalping problem would decrease when the temperature increased. (see Figure 26: Permeation coefficient, Diffusion coefficient and Solubility coefficient of linalool versus Temperature.)

BSR Film (see Table 6): As the temperature was increased from 60 °C to 80 °C, the permeation rate of linalool increased by approximately 1.94 times (from 14,436 to 27,962 ugms.mil/m².hr). The diffusion rate increased by approximately 4.12 times (from 0.481 to 1.984 1/hr). However, the solubility rate decreased by approximately 0.47 times (from 30,012.474 to 14,093.75 ugms/m²). The temperature of this test is lower than the previous tests because the flavor can permeate through the BSR film faster than the other films. The permeation rate and the diffusion rate of linalool increased, but the solubility rate decreased when the temperature increased. The result suggested that as the temperature increased, the flavor would permeate through the film more than scalp inside the film. (see Figure 30: Permeation coefficient, Diffusion coefficient and Solubility coefficient of linalool versus Temperature.)

Discussion of Permeation Curves (Figures 7 to 30)

Figure 7, 8, and 9 show the permeation curves of linalool in ABX film at 70 °C, 80 °C, and 90 °C, respectively. ABX film was selected to study how acrylic coated film would perform in terms of acting as an aroma barrier. All of the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 31 shows the permeation coefficient at 35°C. This film has the lowest permeation rate when compared to the others (see Table 7). Figure 32 and 33 show the Transmission Flux curve. The equilibrium state was calculated from $E = 1/2D$; where E is Equilibrium state, and D is diffusion coefficient. To reach the equilibrium state, it would take about 14 days for the ABX film at 35 °C. This film has acrylic coating on both sides of the film. The result showed that acrylic might be a good aroma barrier.

Figure 11, 12, and 13 show the permeation curves of linalool in HBS film at 70 °C, 80 °C, and 90 °C, respectively. This film, HBS, was selected to study how PVdC coated film would perform in terms of acting as an aroma barrier. All of the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 34 shows the permeation coefficient at 35 °C. This film has a higher permeation rate than ABX although the ABX is 0.05 mils thicker than the HBS. Moreover, the ABX is a two-side acrylic-coated film but the HBS is only a one-side PVdC-coated film (see Table 7). Figure 35 and 36 show the Transmission Flux curve. To reach the equilibrium state, it would take about 4 days for the HBS film at 35 °C. This film has PVdC coated only on one side of the film. Therefore, the PVdC-coated film has a lower aroma barrier than the two-side acrylic-coated film.

Figure 15, 16, and 17 show the permeation curves of linalool in 60 MAC film at 70 °C, 80 °C, and 90 °C, respectively. The 60 MAC film was selected to study how acrylic coated and metallized film would perform in terms of acting as an aroma barrier. All the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 37 shows the permeation coefficient at 35 °C. This film has a higher permeation rate than ABX and HBS (see Table 7). Figure 38 and 39 show the Transmission Flux curve. To reach the equilibrium state, it would take about 6 days for the 60 MAC film at 35°C. Once again, the result confirms that acrylic coating film might help for aroma barrier even if it is only a one-side coated film.

Figure 19, 20, and 20 show the permeation curves of linalool in MET-HB film at 70 °C, 80 °C, and 90 °C, respectively. MET-HB film was selected to study how metallized film would perform in terms of acting as an aroma barrier. All of the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 40 shows the permeation coefficient at 35 °C. This film has a higher permeation rate than ABX, 60 MAC, and HBS (see Table 7). Figure 41 and 42 show the Transmission Flux curve. To reach the equilibrium state, it would take about a half day for the MET-HB film at 35 °C. The solubility coefficient of MET-HB is lower than the 60 MAC, but the permeation coefficient and diffusion coefficient are higher. This means that the acrylic-coated film helps for permeation and diffusion protection, but the metallizing helps for solubility protection. The quality of barrier properties depends on the quality of the film, for instance, pin-holes and unequal metallized layer.

Figure 23, 24, and 25 show the permeation curves of linalool in CONTROL film produced in Thailand, at 70 °C, 80 °C, and 90 °C, respectively. This CONTROL film is used for packing Big Jack, corn snack, presently. The purpose of test-

ing is to know the aroma barrier of CONTROL film, and to compare it to the other films. All of the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 43 shows the permeation coefficient at 35 °C. This film has a higher permeation rate than ABX, 60 MAC, MET-HB and HBS (see Table 7). Figure 44 and 45 show the Transmission Flux curve. To reach the equilibrium state, it would take about 1 day for the CONTROL film at 35 °C.

Figure 27, 28, and 29 show the permeation curves of linalool in BSR film at 60 °C, 70 °C, and 80 °C, respectively. BSR film was selected to study how coextruded film would perform, in terms of acting as an aroma barrier. All of the three permeation curves rose from the baseline steadily, and proceeded up the permeation curve rather rapidly. Figure 46 shows the permeation coefficient at 35°C. This film has a higher permeation rate than ABX, 60 MAC, MET-HB, CONTROL, and HBS even at a lower temperature (see Table 7). Figure 47 and 48 show the Transmission Flux curve. To reach the equilibrium state, it would take about 0.3 of a day for BSR film at 35 °C. This film is coextruded film, and has no chemical coating for aroma barrier.

In Conclusion from Table 7, there was a difference in the barrier property of the six films as compared with linalool. The following are the findings:

1. At 35 °C, the highest permeation rate was BSR film; followed by CONTROL, MET-HB, 60 MAC, HBS and ABX, respectively (see page 108). These results are related to flavor loss.
2. At 35 °C, the highest diffusion rate was also BSR film; followed by MET-HB, CONTROL, HBS, 60 MAC, and ABX, respectively (see page 108). These results are related to the time it takes to reach equilibrium state and shelf life.

3. At 35 °C, the highest solubility rate was again also BSR film; followed by CONTROL, 60 MAC, MET-HB, HBS, and ABX, respectively (see page 108). These results are related to flavor loss.
4. At 35 °C, the highest equilibrium days was ABX; followed by 60 MAC, HBS, CONTROL, MET-HB and BSR, respectively (see page 109).

Conclusion

From all data, the conclusion could be drawn as follows:

1. Among the three non-metallized films (ABX, HBS, and BSR), ABX has the best flavor barrier while BSR is worst at 35 °C. HBS (PVdC coated film) is known as a good WVTR barrier film. However, HBS does not have a better flavor barrier than ABX. Therefore, a good WVTR film is not necessarily a good flavor barrier film.
2. Among the three metallized films, the 60 MAC, acrylic coated on one side, seems to give a highest aroma barrier. The CONTROL film, Thai film, was the second highest for aroma barrier, and the MET-HB was the lowest for aroma barrier.
3. Among all the films, ABX film (an acrylic coated film) yields the most aroma barrier property at 35 °C. It would also be suitable to be used for flavor barrier packaging. Since flavor loss is a combination of amount permeated and amount scalped, ABX film is the best in both cases.
4. The BSR (coextrusion film) would not be recommended for barrier property purpose film because it has high permeation rate.
5. The ABX, HBS and 60 MAC would be used instead of the CONTROL film to extend the shelf life of the Big Jack, corn snack.

4. The BSR, coex film, would not be recommended for barrier property purpose film because it has the highest permeation rate.
5. The ABX, HBS and 60 MAC would be used instead of the CONTROL film to extend the shelf life of Big Jack, corn snack.

Recommendations for Further Research

The sensory evaluation test of the product packaged in three recommended films in this study, will give more reliable results before the change of the packaging material is decided. Furthermore, the other compounds in the Spicy Chicken flavor, besides linalool, could also be tested.

References

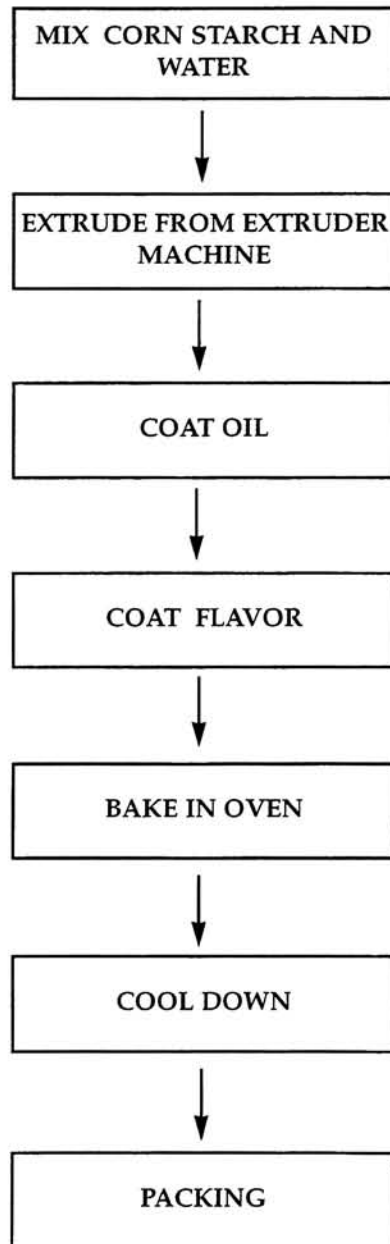
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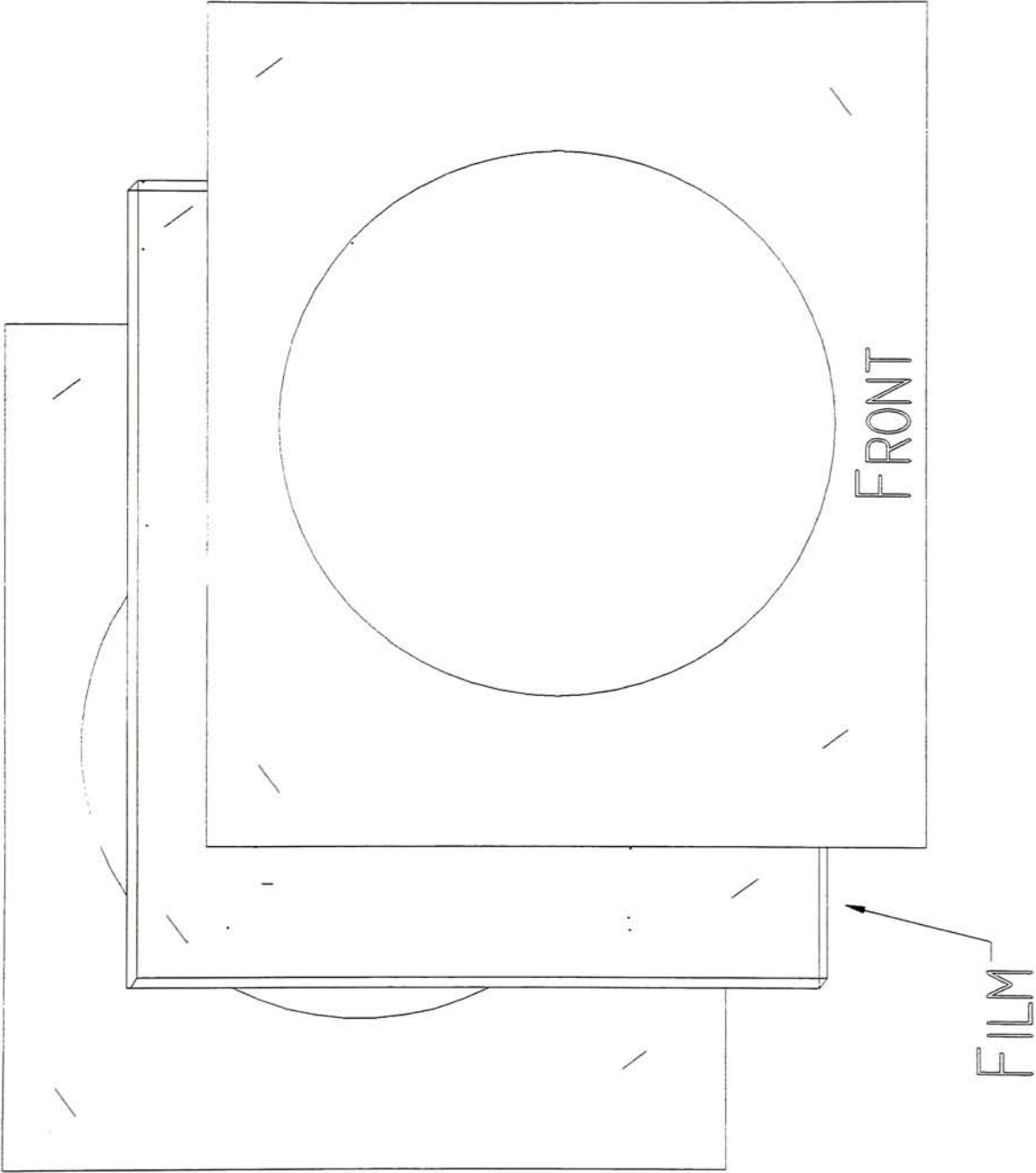
Appendix A

PROCESS FLOW CHART OF CORN SNACK



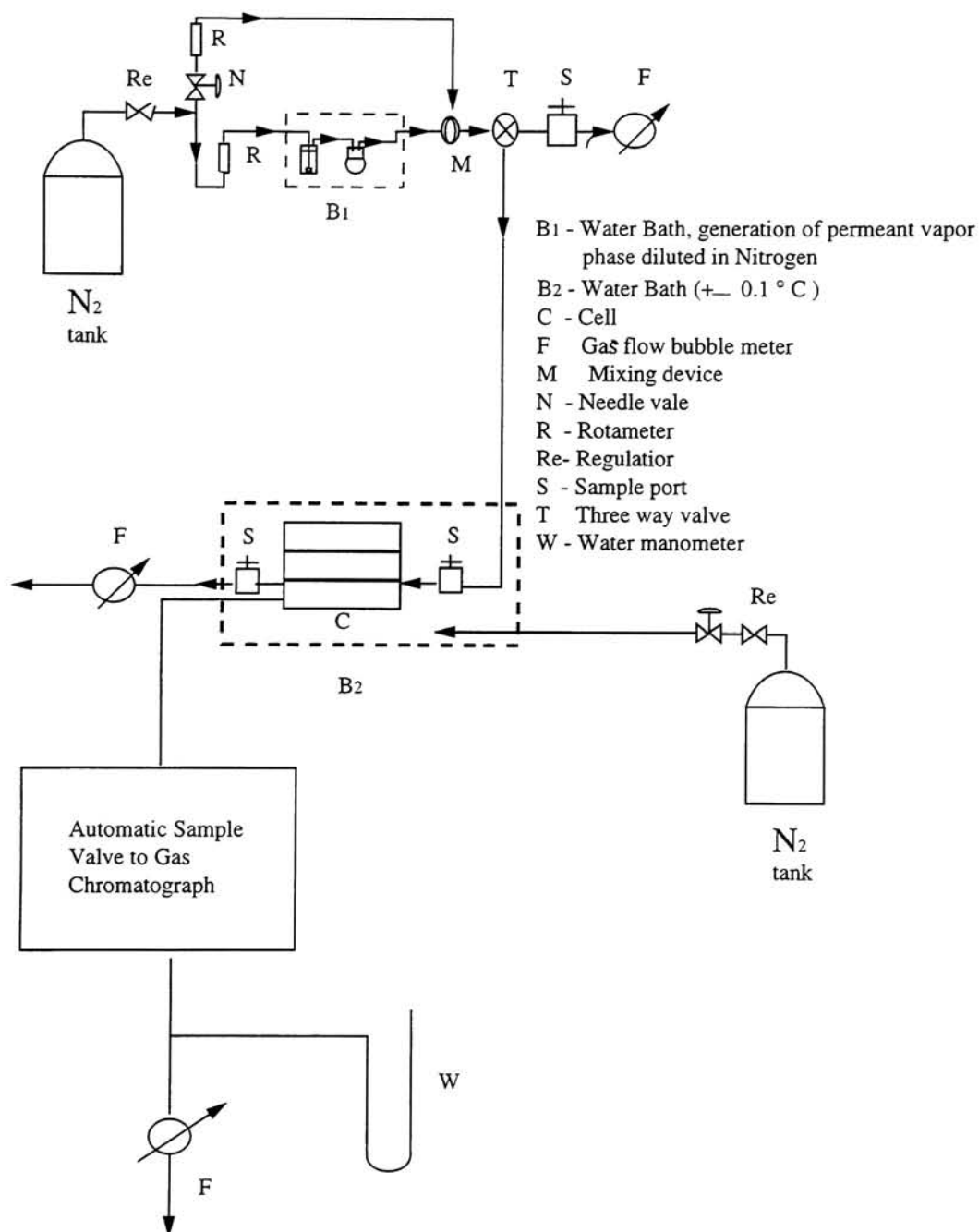
Appendix B

Sample Preperation



Appendix C

Schematic Diagram of the Isostatic Test Apparatus



MAS2000TM

Isostatic Organic Diffusion Apparatus

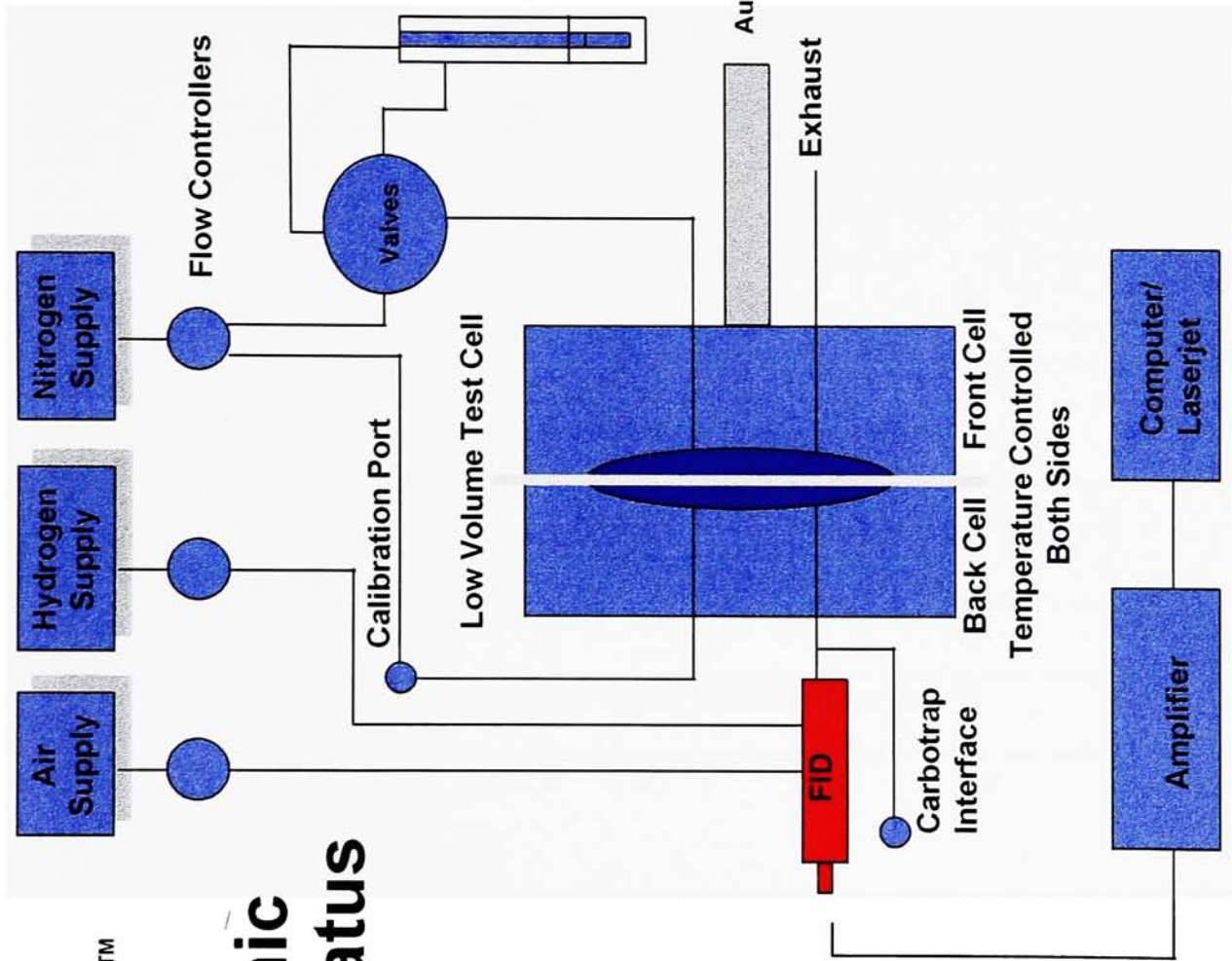


Table 1 Permeation Characteristics of linalool in ABX Film

From Figure 7-10

Temperature	70°C	80°C	90°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	182	725	2635	14.47802198
Diffusion (1/hr)	0.08	0.211	0.552	6.9
Solubility (ugms/m2)	2275	3436.01896	4773.55072	2.098264055

Table 2 Permeation Characteristics of linalool in HBS Film

From Figure 11-14

Temperature	70°C	80°C	90°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	189	563	1165	6.164021164
Diffusion (1/hr)	0.186	0.478	1.049	5.639784946
Solubility (ugms/m2)	1016.12903	1177.82427	1110.58151	1.092953228

Table 3 Permeation Characteristics of linalool in 60 MAC Film

From Figure 15-18

Temperature	70°C	80°C	90°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	608	1427	2713	4.462171053
Diffusion (1/hr)	0.114	0.341	0.638	5.596491228
Solubility (ugms/m2)	5333.33333	4184.75073	4252.3511	0.797315831

Table 4 Permeation Characteristics of linalool in MET-HB Film

From Figure 19-22

Temperature	70°C	80°C	90°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	373	640	527	1.412868633
Diffusion (1/hr)	0.592	1.163	2.156	3.641891892
Solubility (ugms/m2)	630.067568	550.300946	244.434137	0.387949087

Table 5 Permeation Characteristics of linalool in CONTROL Film

From Figure 23-26

Temperature	70°C	80°C	90°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	2150	5283	4686	2.179534884
Diffusion (1/hr)	0.182	0.217	0.475	2.60989011
Solubility (ugms/m2)	11813.1868	24345.6221	9865.26316	0.835105998

Table 6 Permeation Characteristics of linalool in BSR Film

From Figure 27-30

Temperature	60°C	70°C	80°C	Percent of Changing
Permeation (ugms.mil/m2.hr)	14436	19455	27962	1.936963148
Diffusion (1/hr)	0.481	1.431	1.984	4.124740125
Solubility (ugms/m2)	30012.474	13595.3878	14093.75	0.469596408

Table 7 Permeation Characteristics of linalool in Six Film at 35°C

Film Type	Perm.	Diff.	Sol.	Equilibrium(days)
ABX	0.73	0.001	503	14.29
HBS	4.67	0.005	880	3.93
60 MAC	28.59	0.004	8113	5.91
MET-HB	202.88	0.041	4894	0.5
CONTROL	497.35	0.023	21688	0.91
BSR	5527.45	0.07	78517	0.3

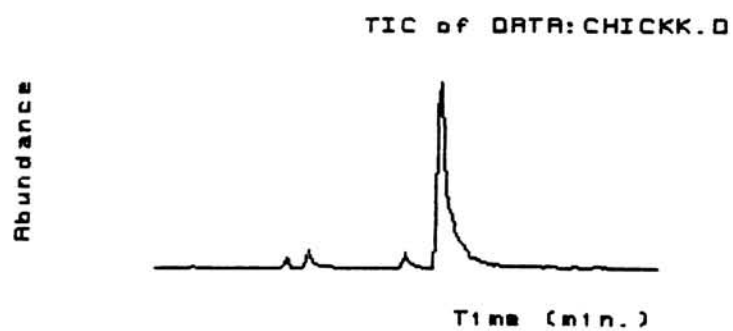
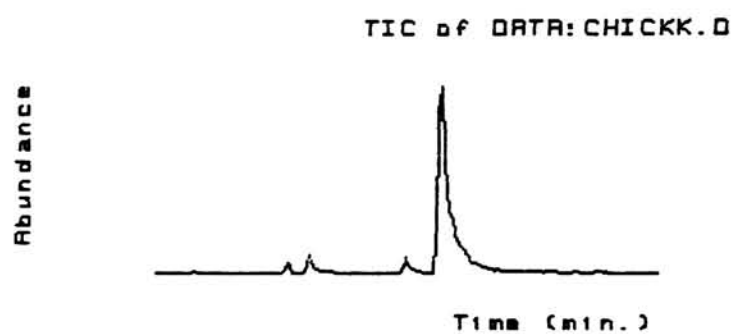


Figure 1

	actual	Setpt	Limit		actual	Setpt	Limit
Oven (Standby)	94	100	300	Inj Port	250	250	275
Transfer Line	206	200	220	Ion Source	200	200	220
Mass Analyzer	260	260	275	FID	72	Off	0

58

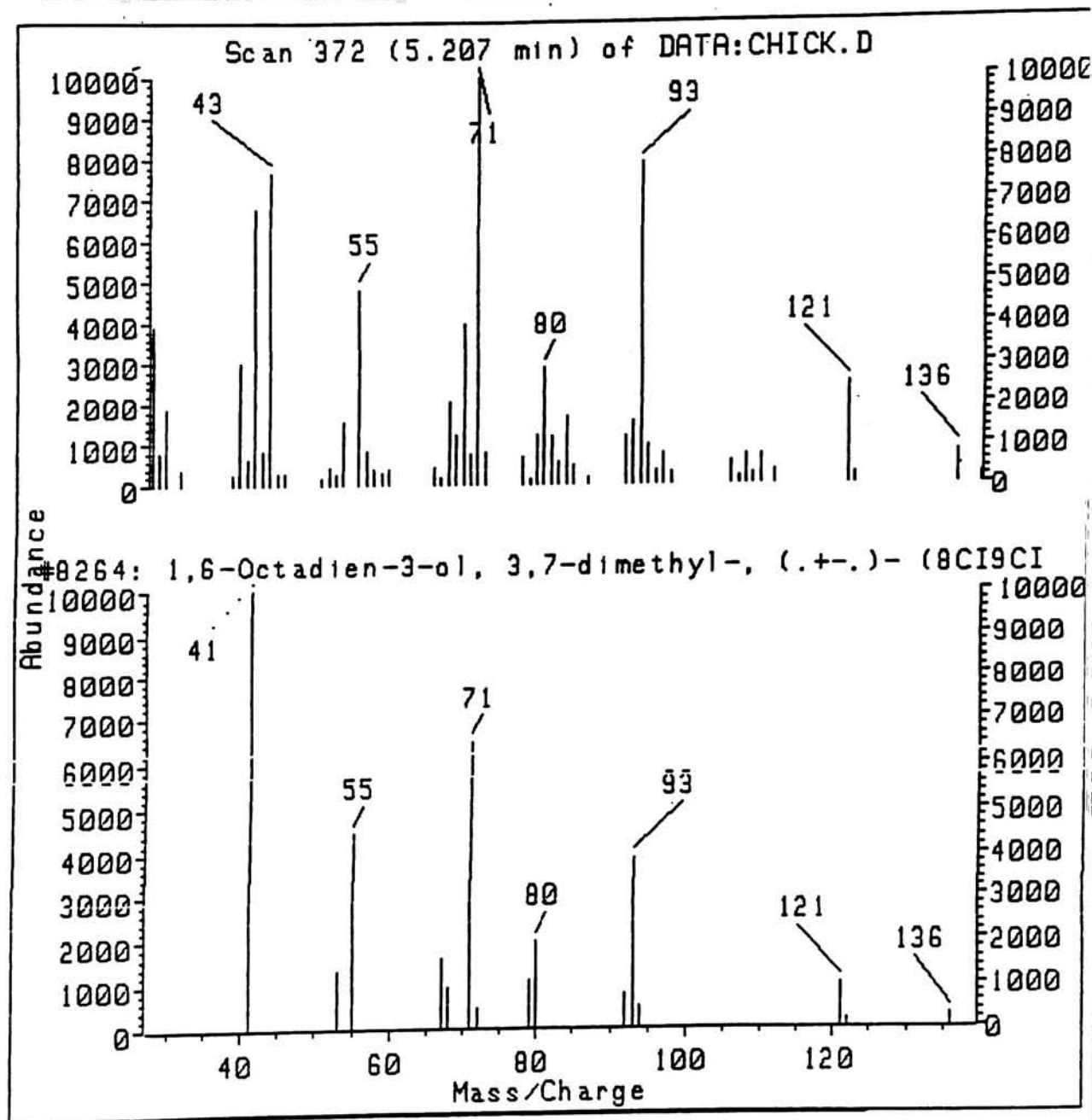
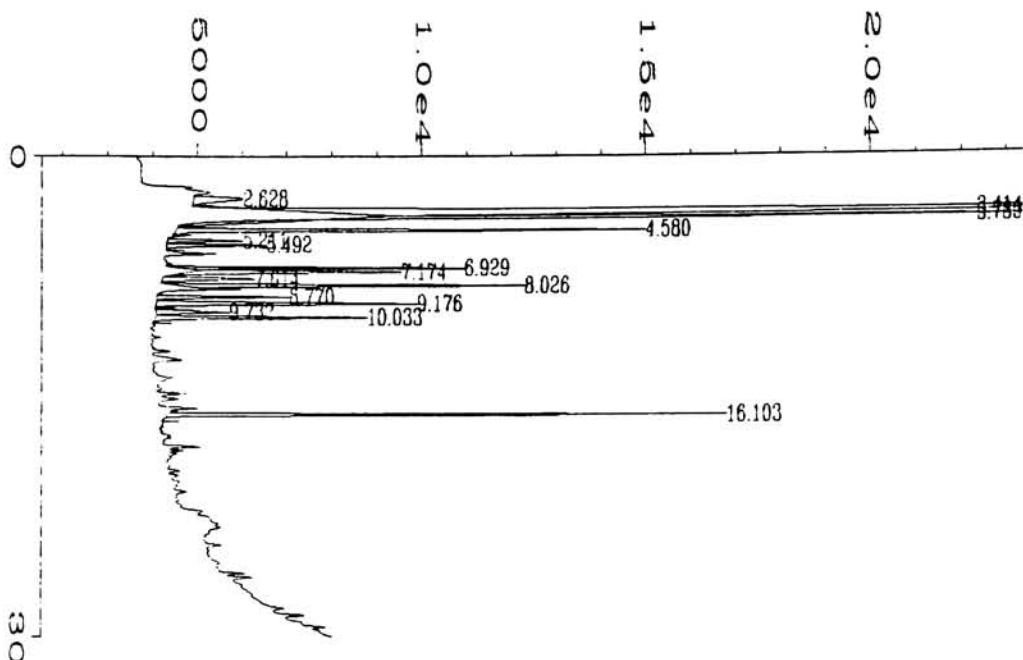


Figure 2



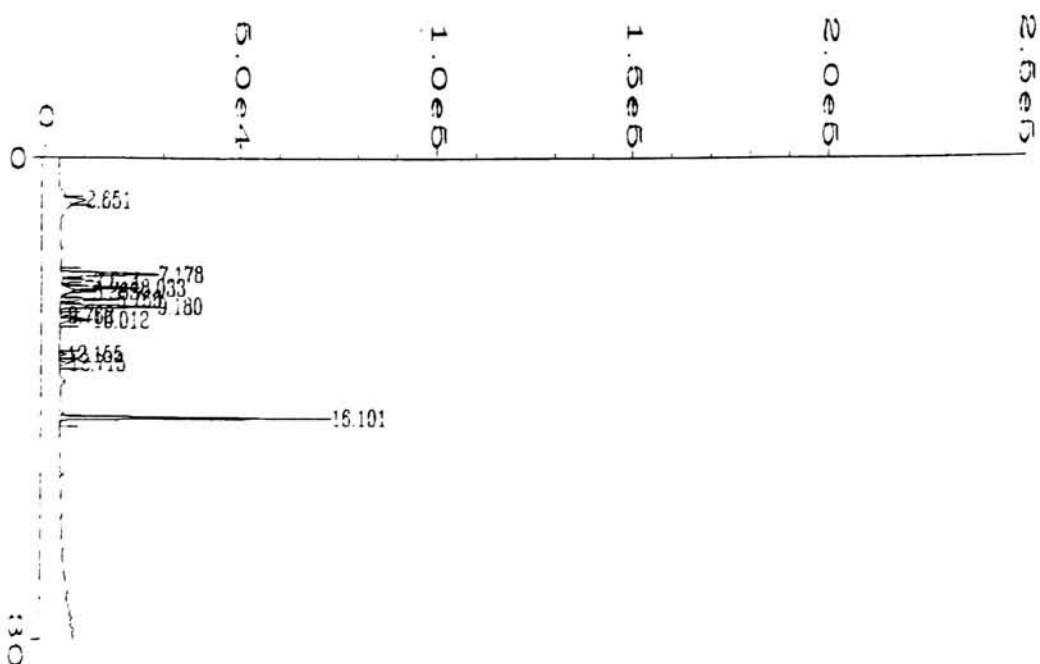
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 Area Percent Report
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 Instrument : GC 1 Vial Number : 2
 Sample Name : various samples Injection Number :
 Run Time Bar Code: Sequence Line :
 Acquired on : 09 Jan 96 04:57 PM Instrument Method: DESORB6.MTH
 Report Created on: 09 Jan 96 05:31 PM Analysis Method : DESORB6.MTH

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2	2.868	0	295	Rsho	0.000	0.0000
3	3.125	0	-430	Fsho	0.000	0.0000
4	3.414	380609	27293	BB	0.226	39.6549
5	3.518	0	15880	Rsho	0.000	0.0000
6	3.780	83441	17847	BB	0.079	8.6936
7	4.580	76702	10500	BB	0.114	7.9914
8	5.211	12271	1703	BB	0.119	1.2785
9	5.492	16235	2252	BB	0.107	1.6915
10	6.929	38835	6223	BB	0.100	4.0462
11	7.174	46489	4734	BB	0.147	4.8436
12	7.614	18621	2058	BB	0.143	1.9401
13	8.026	60389	7634	BB	0.126	6.2918
14	8.770	21006	2960	BB	0.111	2.1886
15	9.176	54892	5834	BB	0.143	5.7191
16	9.692	0	1225	Fsho	0.000	0.0000
17	9.732	17027	1616	BB	0.151	1.7740
18	10.033	35864	4717	BB	0.116	3.7366
19	16.103	77580	12521	BB	0.097	8.0329

Figure 4



Area Percent Report

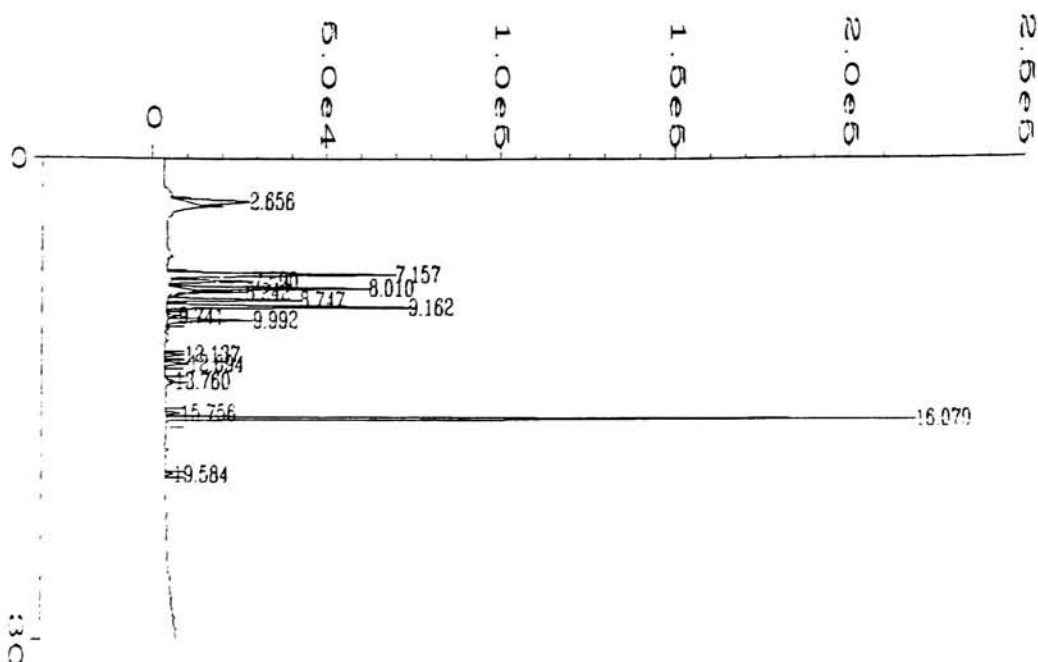
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4	7.614	72264	7920	BB	0.144	5.0204
5	8.033	138786	17991	BB	0.124	9.6419
6	8.265	23130	5240	BB	0.081	1.6069
7	8.766	100809	14062	BB	0.113	7.0035
8	9.180	211696	24718	BB	0.132	14.7072
9	9.762	13066	1765	BB	0.118	0.9077
10	10.012	67573	8220	BB	0.125	4.6945
11	10.161	0	1270	Rsho	0.000	0.0000
12	12.155	16829	1976	BB	0.133	1.1692
13	12.715	23249	2527	BB	0.138	1.6152
14	16.101	432658	69286	BB	0.098	30.0582

Total area 1439402

Figure 5



Area Percent Report

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3	7.291	0	21549	Rsho	0.000	0.0000
4	7.590	203440	23515	BB	0.134	4.9153
5	8.010	395417	52614	BB	0.118	9.5537
6	8.242	71576	15286	BB	0.082	1.7293
7	8.747	265189	37890	BB	0.109	6.4072
8	9.162	571778	69497	BB	0.125	13.8147
9	9.601	0	278	Fsho	0.000	0.0000
10	9.741	22110	2989	BB	0.116	0.5342
11	9.992	192142	24258	BB	0.120	4.6423
12	10.140	0	3281	Rsho	0.000	0.0000
13	12.137	47525	5626	BB	0.133	1.1482
14	12.694	56814	6735	BB	0.128	1.3727
15	13.700	10910	1859	BB	0.095	0.2636
16	15.756	28109	4184	BB	0.106	0.6791
17	16.079	1323238	215694	BB	0.097	31.9708
18	19.584	17150	2411	BB	0.112	0.4144

Total area 4108829

Figure 6

material:	ABX	permeant:	linalool	cell temp:	70
caliper:	.75	concentration:	1	fid temp:	150

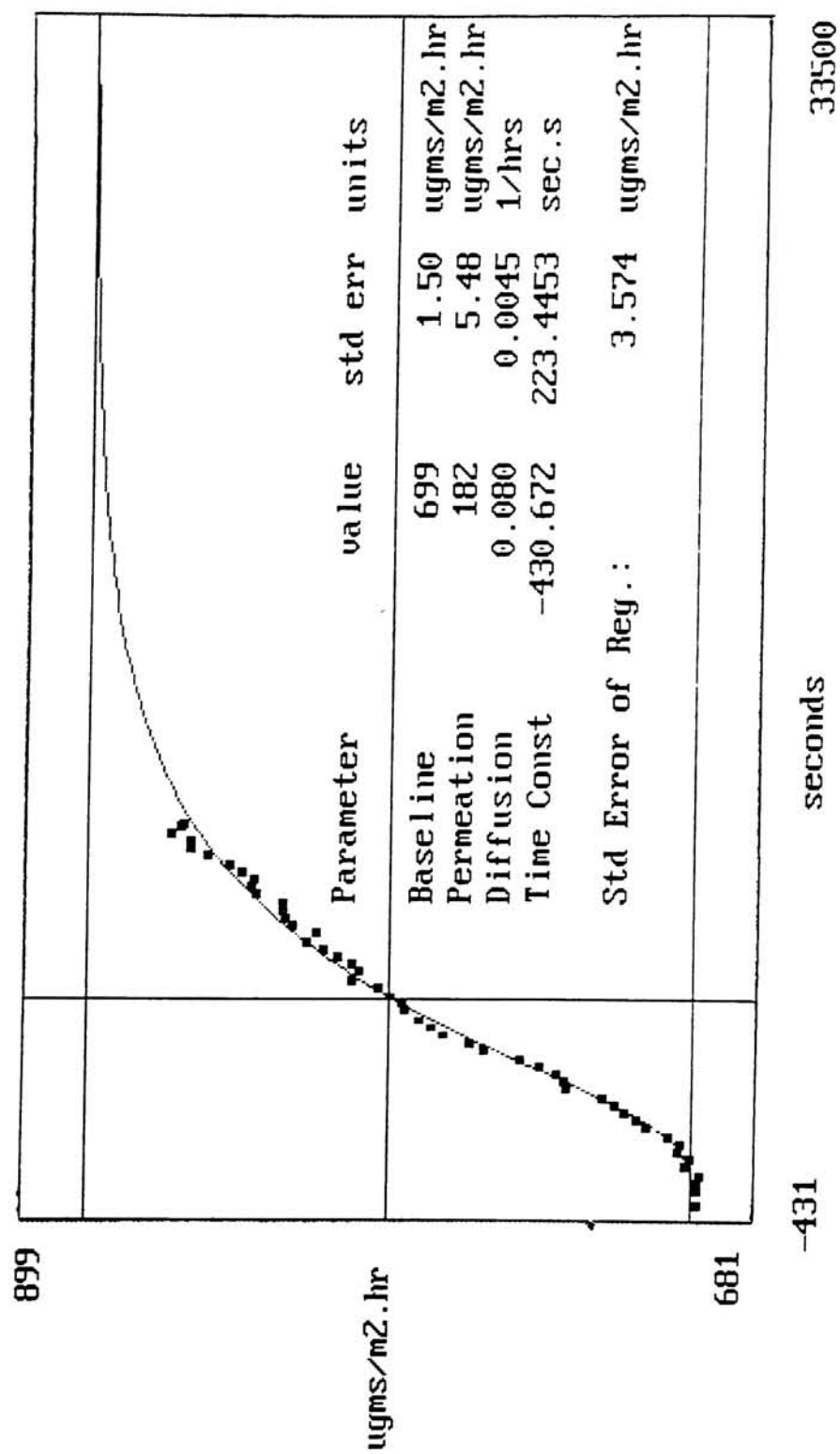
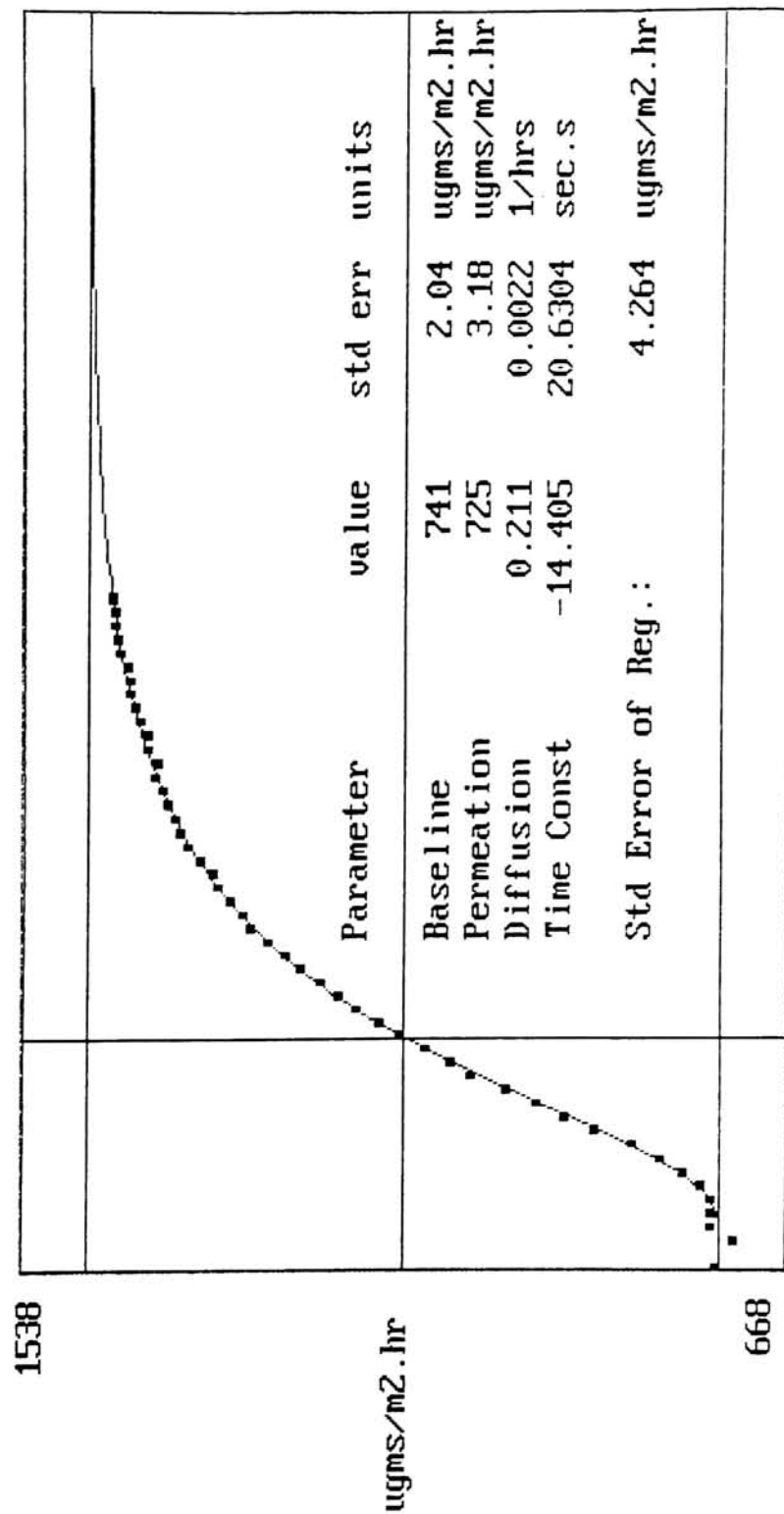


Figure 7

material: ABX
caliper: .75

permeant: linalool
concentration: 1
cell temp: 80
fid temp: 150



-14

seconds

12771

f1:save f2:report setup f3:exit

Figure 8

material: ABX
caliper: .75

permeant: linalool
concentration: 1
cell temp: 90
fid temp: 150

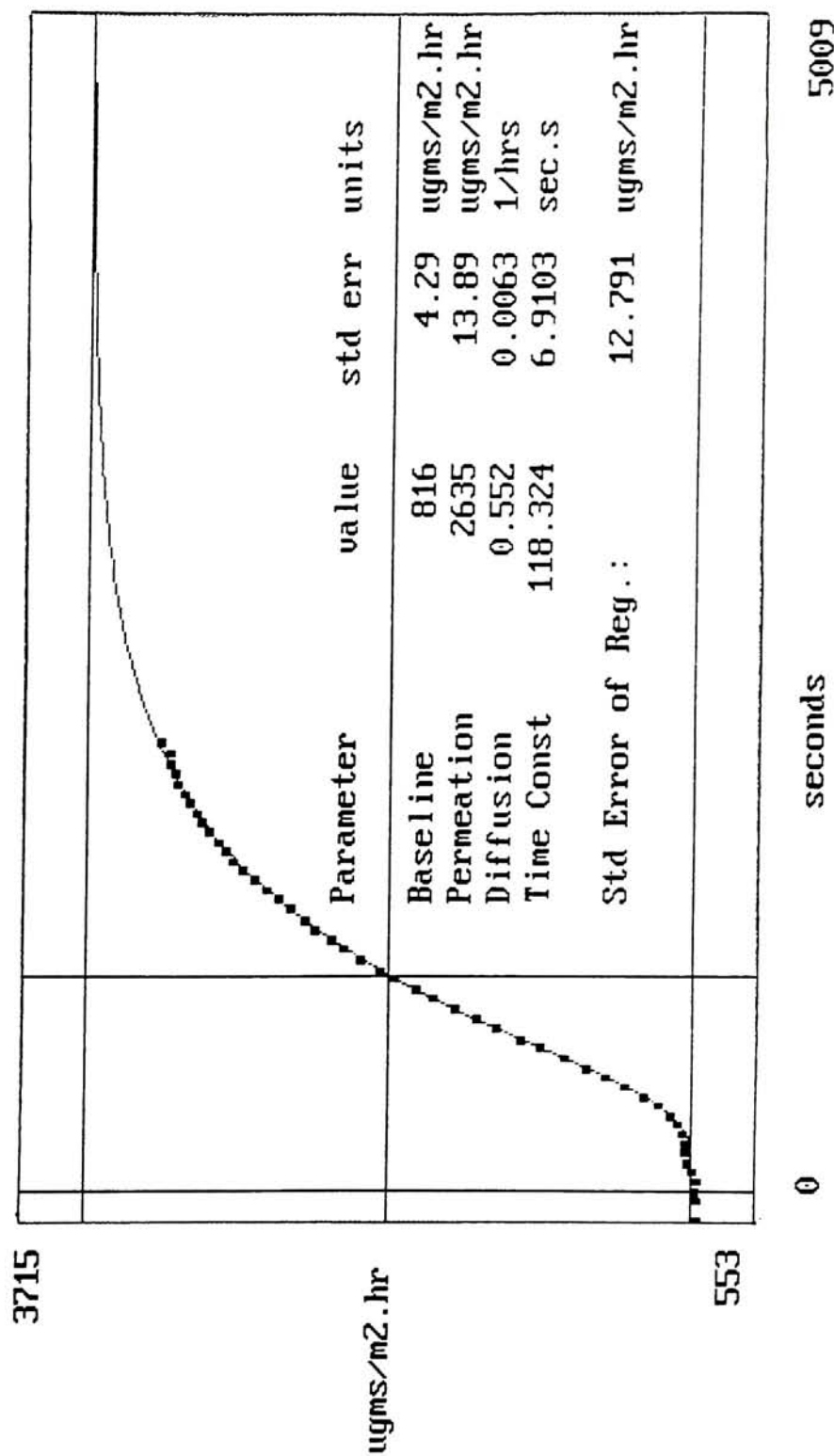


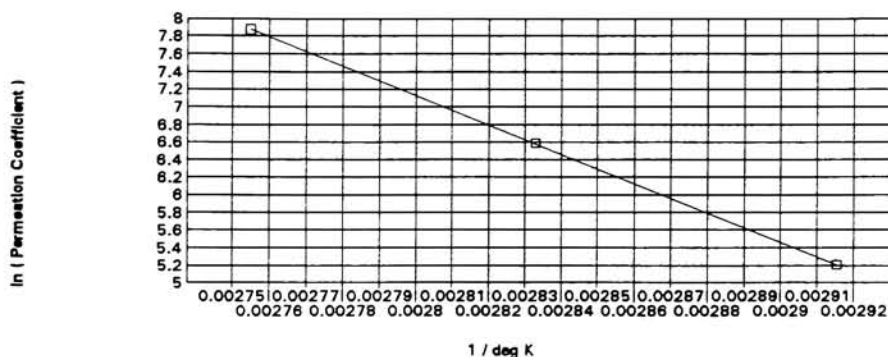
Figure 9

Material:	ABX
Caliper (mils):	0.75

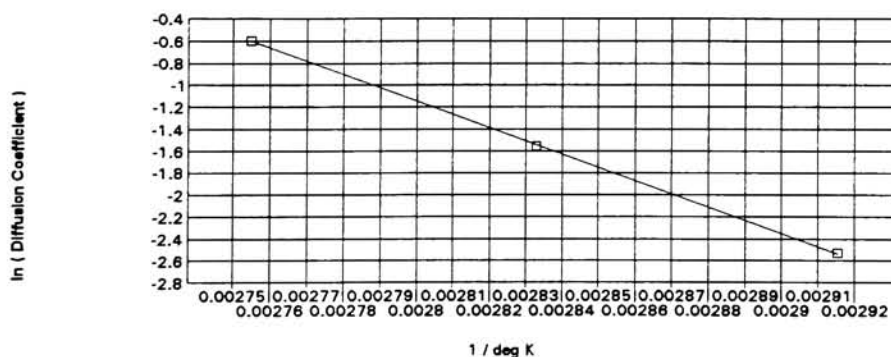
Permeant:	linalool
Concentration:	1.00

Permeation Units:	ugms/m2.hr
Diffusion Units:	1/hrs
Solubility Units:	ugms/m2

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

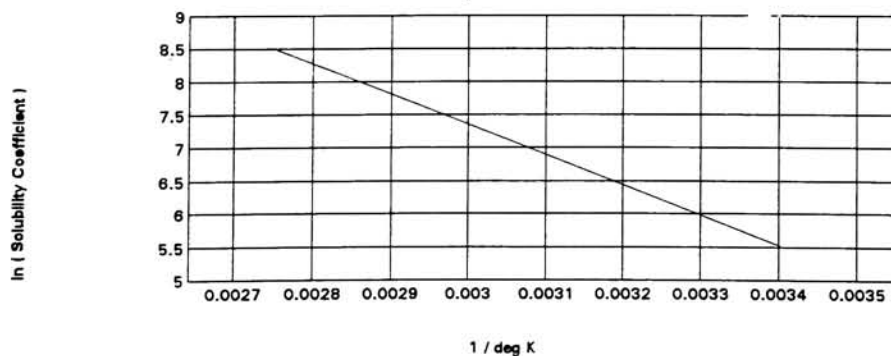


Figure 10

material: HBS
caliper: .7

permeant: linalool
concentration: 1
cell temp: 70
fid temp: 150

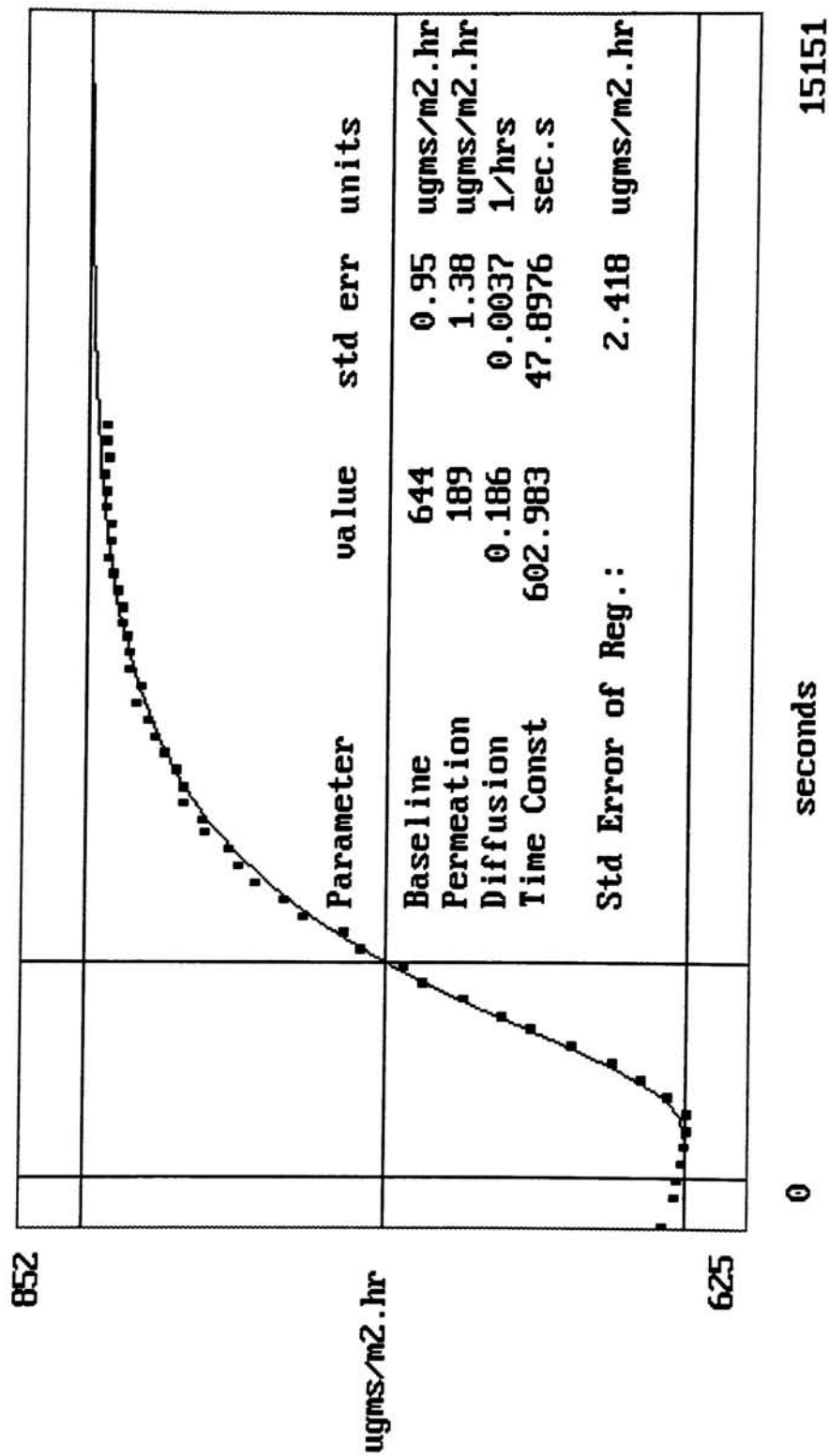


Figure 11

material: HBS
caliper: .7

permeant: linalool
concentration: 1

cell temp: 80
fid temp: 150

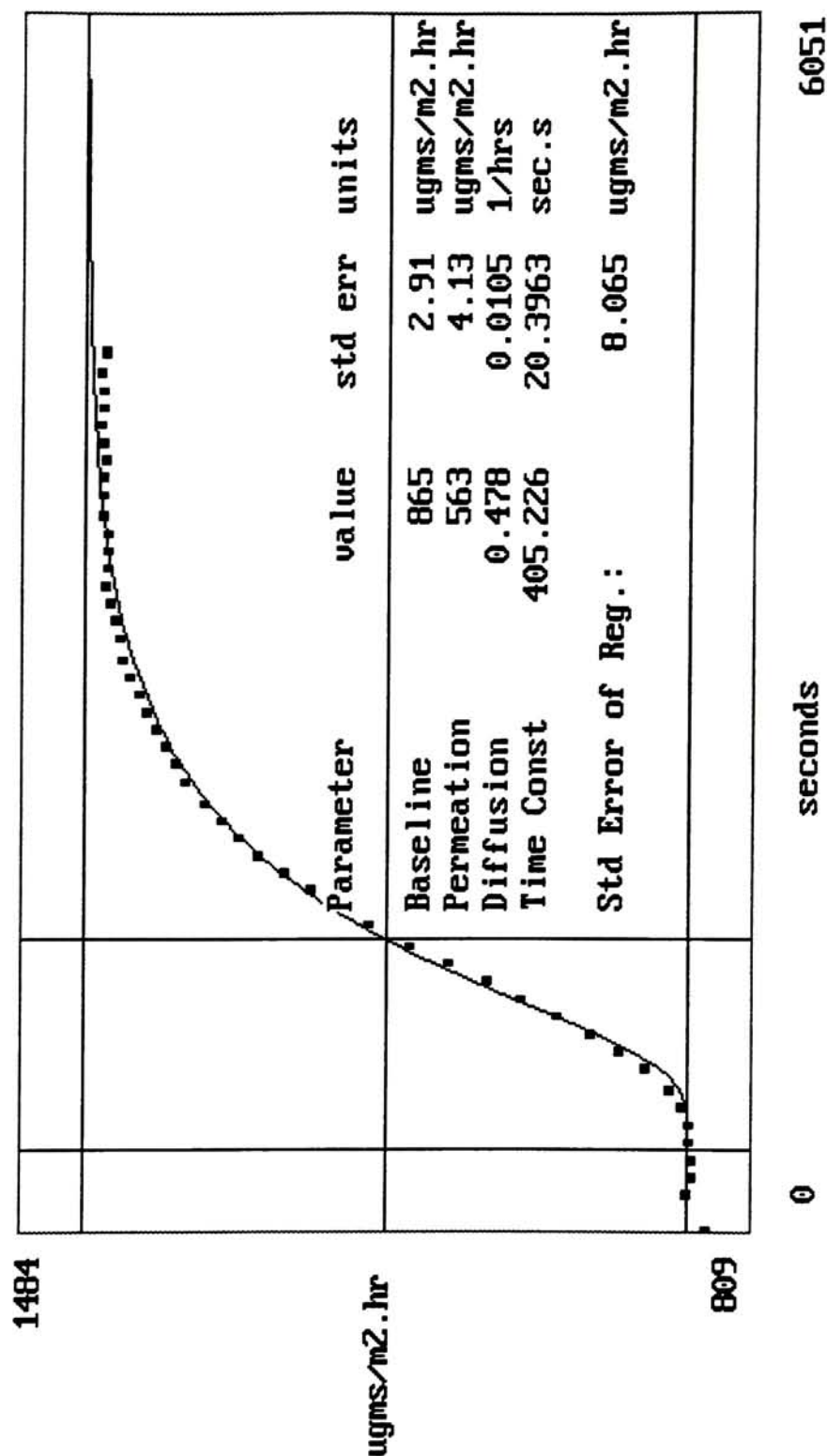


Figure 12

material: HBS
caliper: .7

permeant: linalool
concentration: 1
cell temp: 90
fid temp: 150

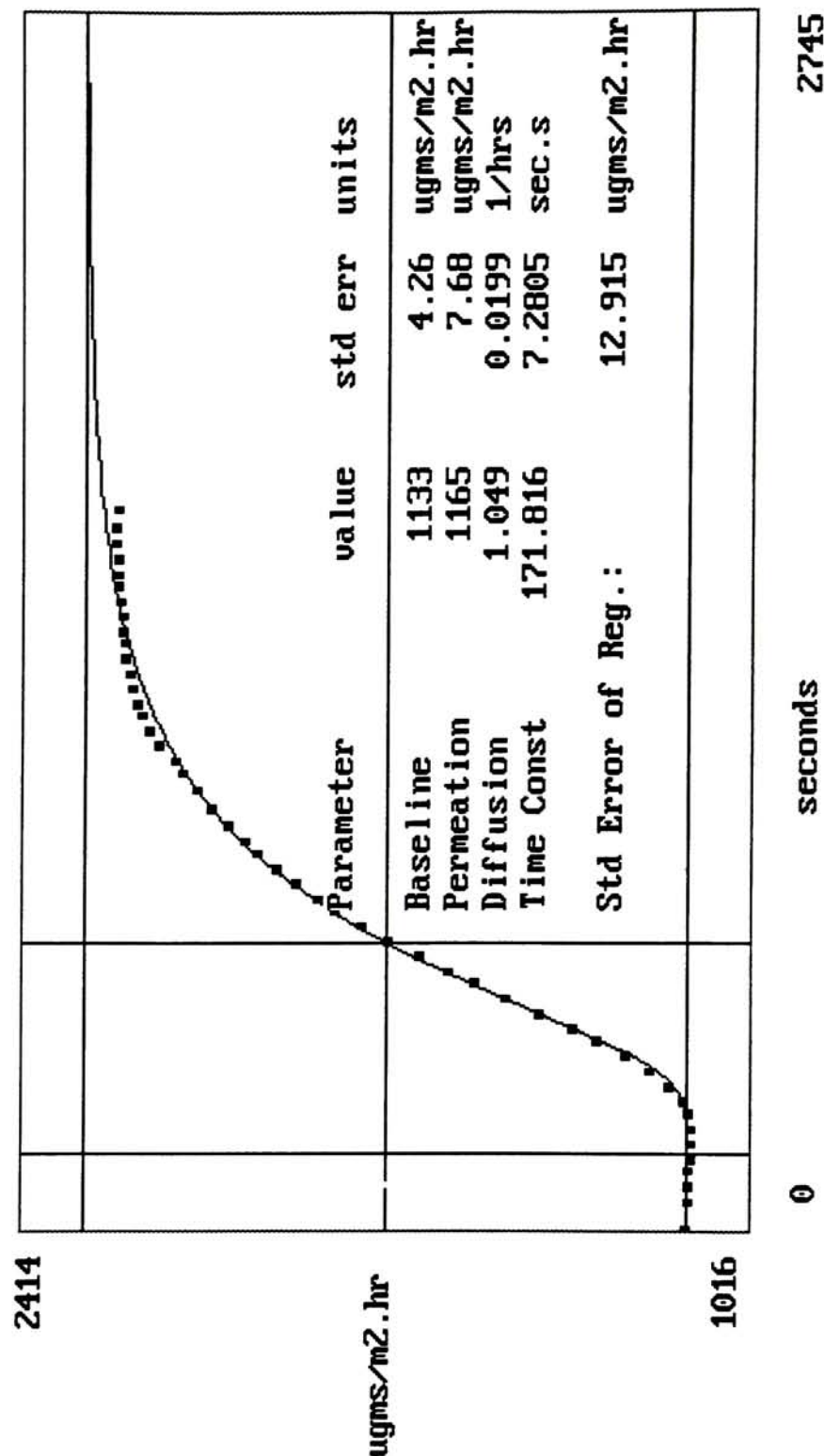


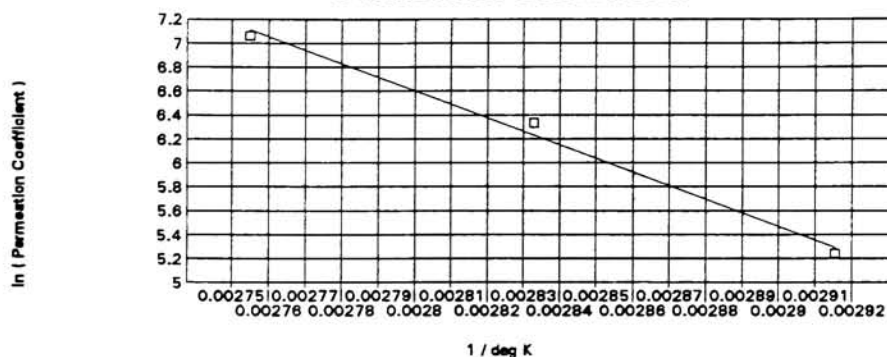
Figure 13

Material: **HBS**
Caliper (mils): **0.7**

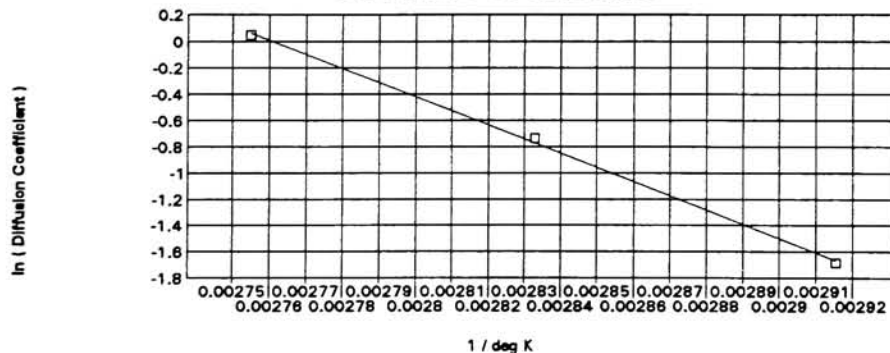
Permeant: **linalool**
Concentration: **1.00**

Permeation Units: **ugms/m2.hr**
Diffusion Units: **1/hrs**
Solubility Units: **ugms/m2**

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

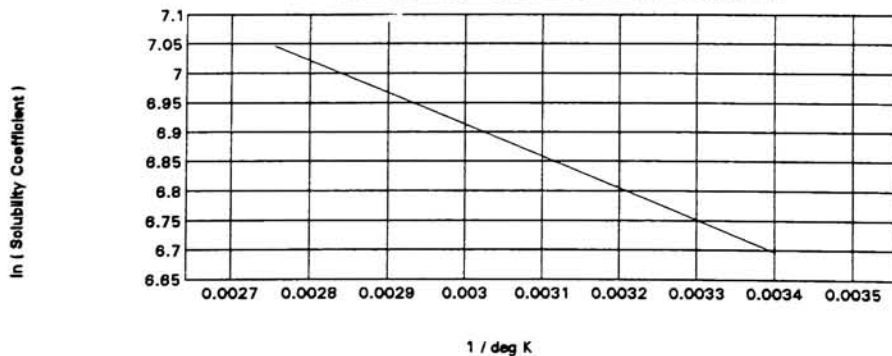
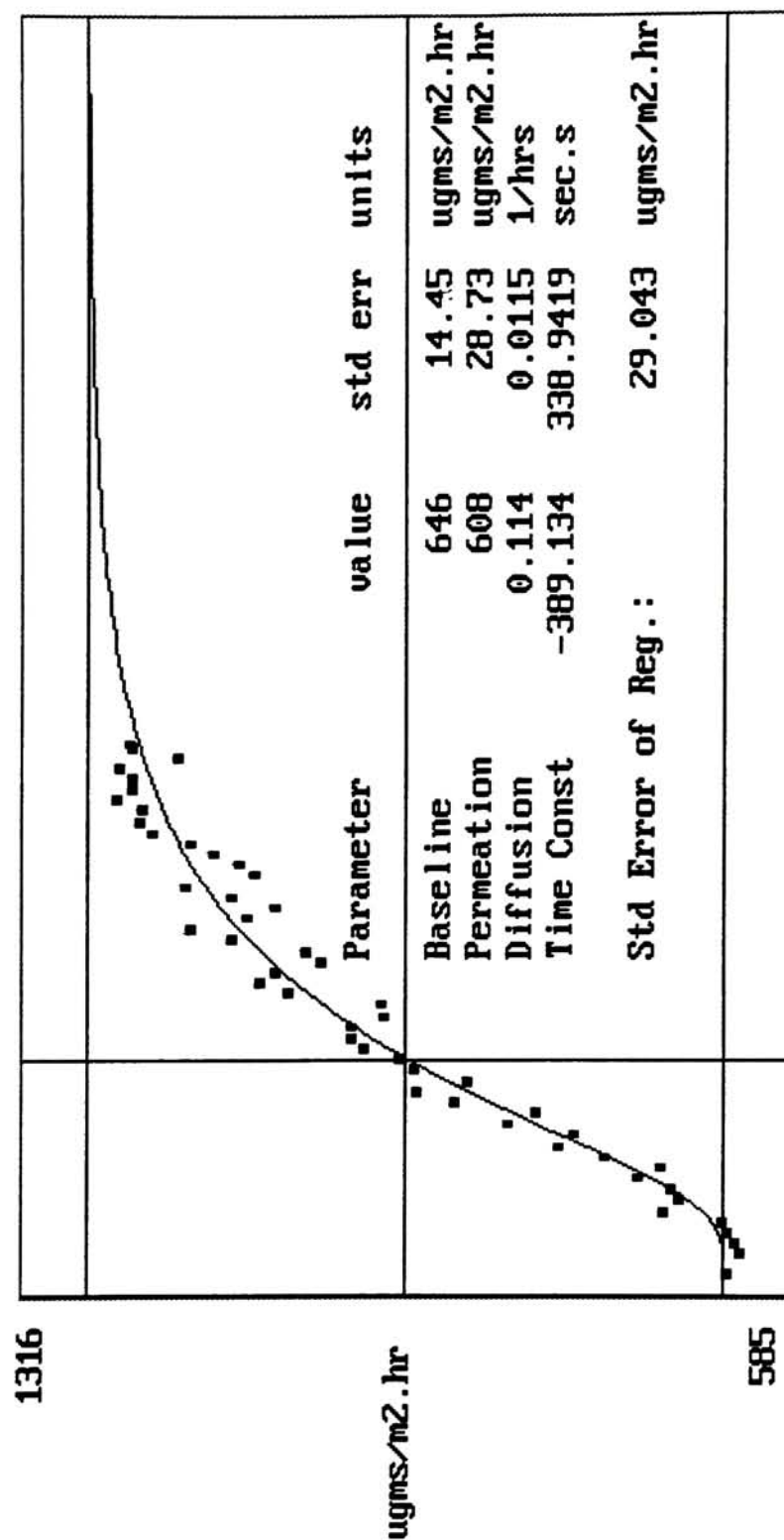


Figure 14

material: 60 MAC
 caliper: .6
 permeant: linalool
 concentration: 1
 cell temp: 70
 fid temp: 150



-389

seconds

23192

f1:save f2:report setup f3:exit

Figure 15

material: 60 MAC
caliper: .6

permeant: linalool
concentration: 1
cell temp: 80
fid temp: 150

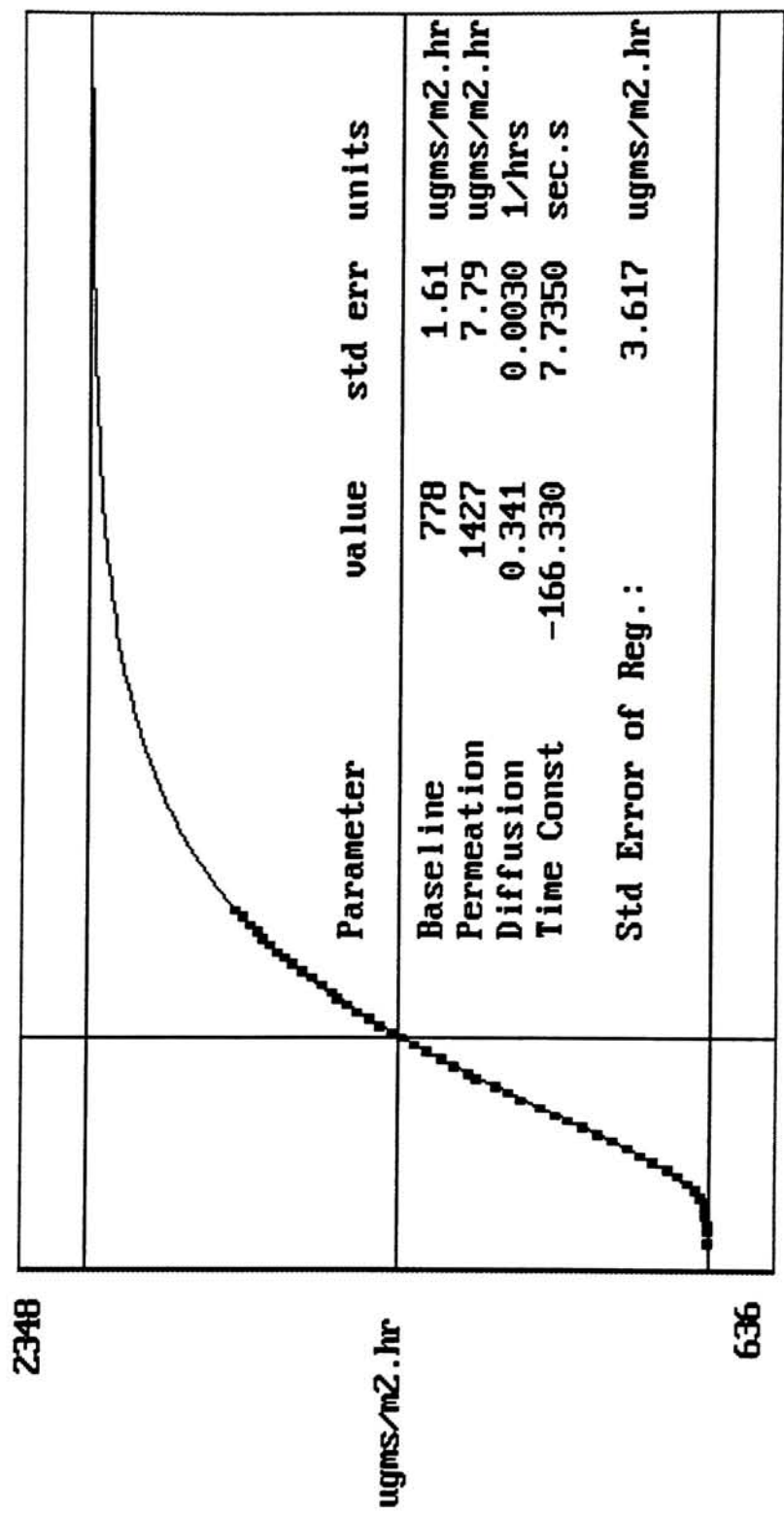


Figure 16

material: 60 MAC
caliper: .6

permeant: linalool
concentration: 1
cell temp: 90
fid temp: 150

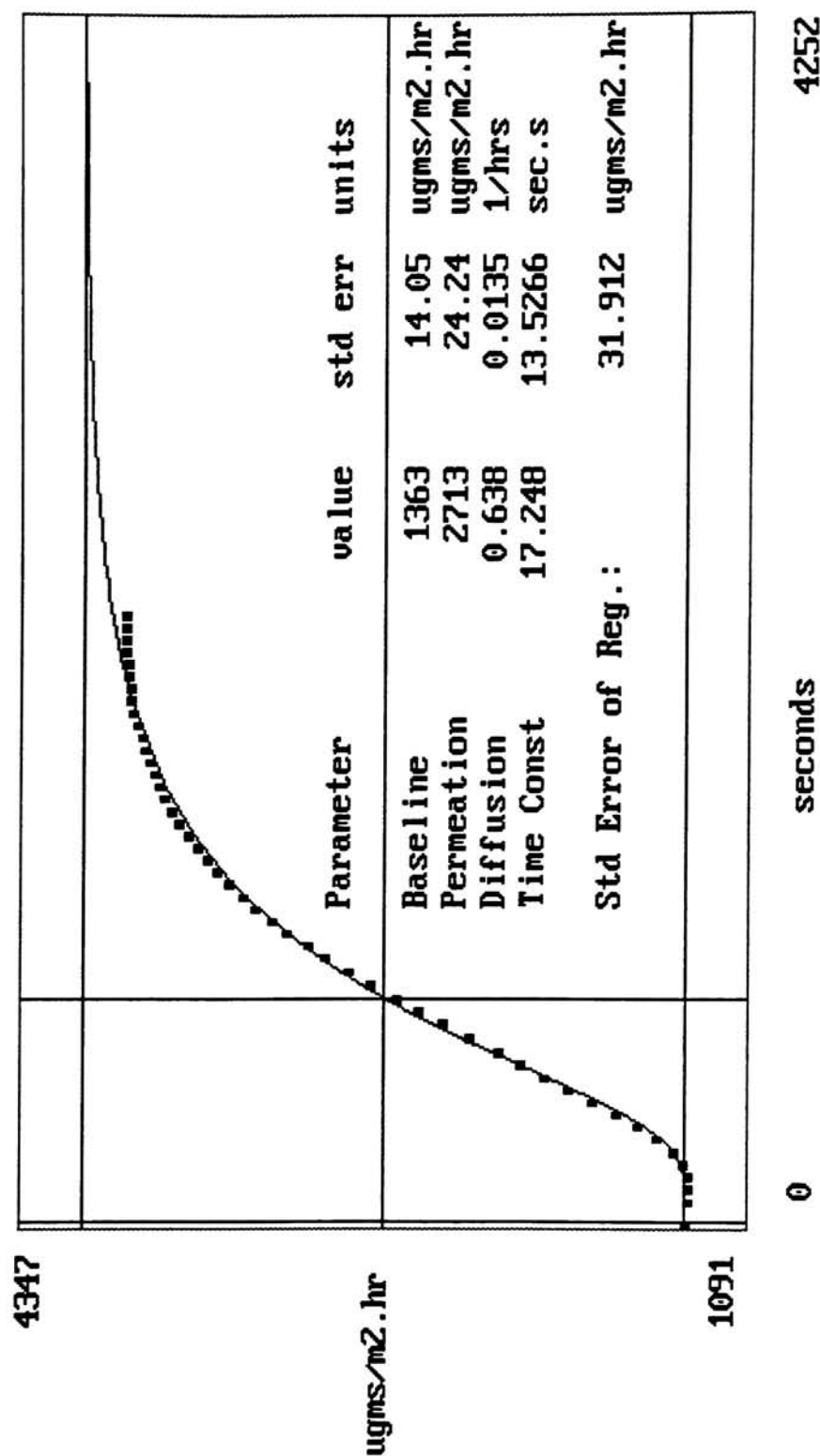


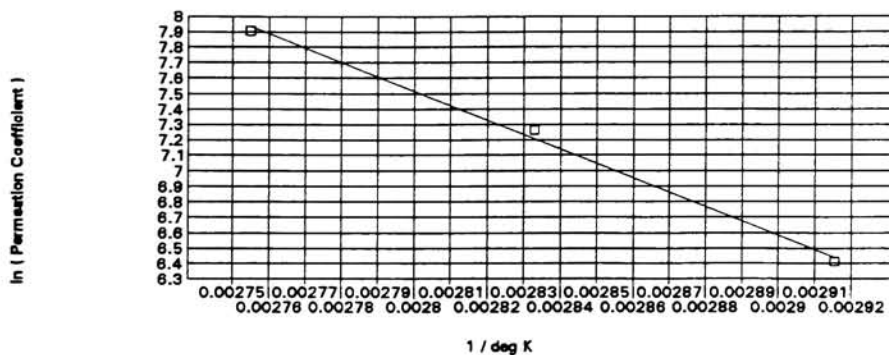
Figure 17

Material: **60 MAC**
Caliper (mils): **0.6**

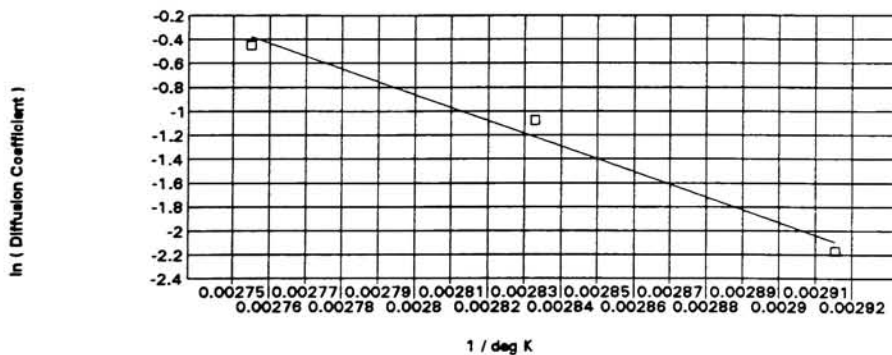
Permeant: **linalool**
Concentration: **1.00**

Permeation Units: **ugms/m2.hr**
Diffusion Units: **1/hrs**
Solubility Units: **ugms/m2**

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

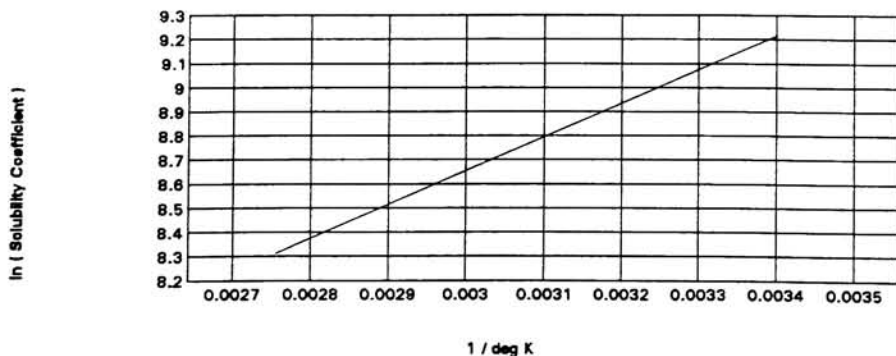


Figure 18

material: Met-HB
caliper: .7

permeant: linalool
concentration: 1

cell temp: 70
fid temp: 150

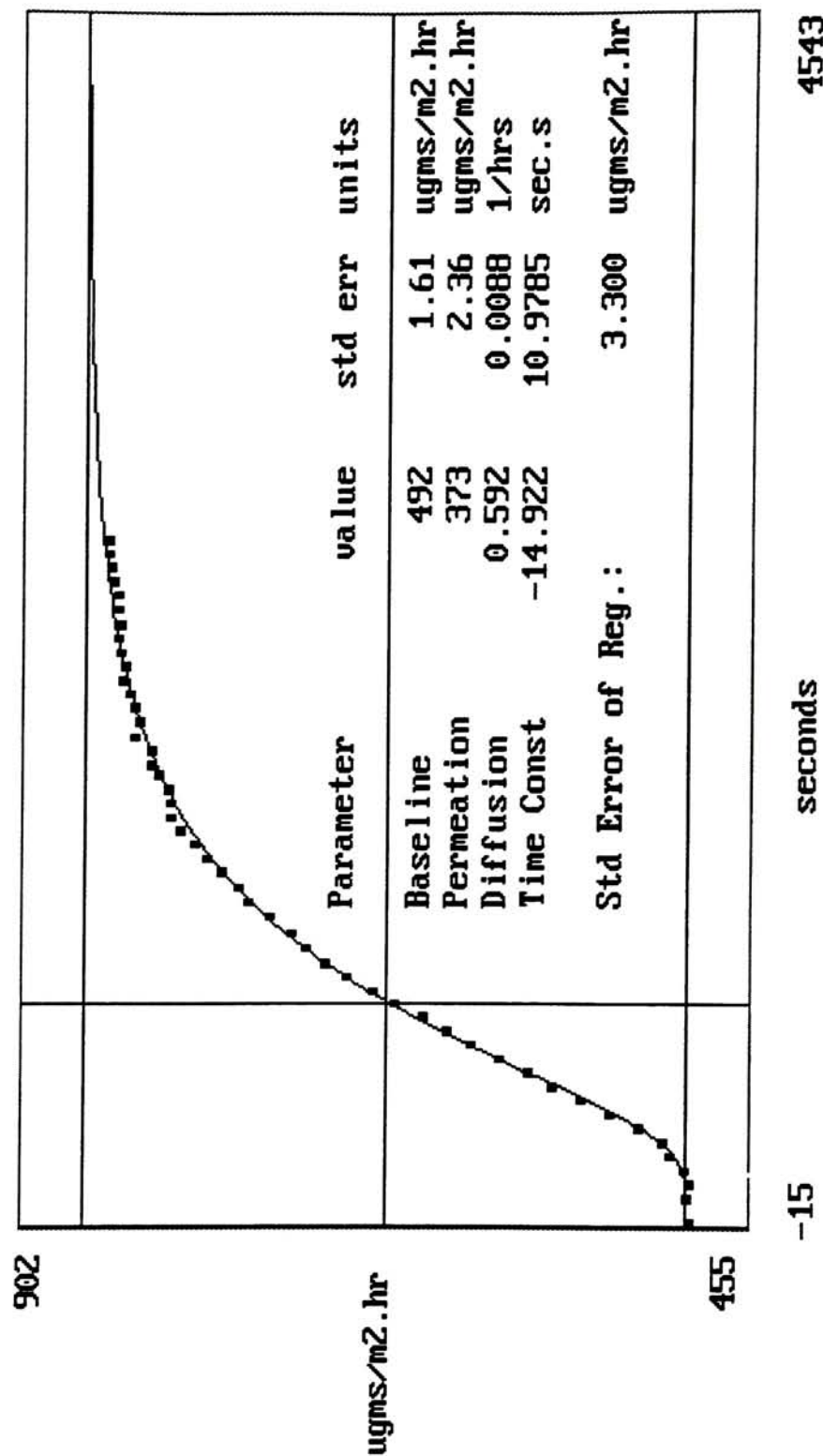


Figure 19

material: Met-HB
 caliper: .7
 permeant: linalool
 concentration: 1
 cell temp: 80
 fid temp: 150

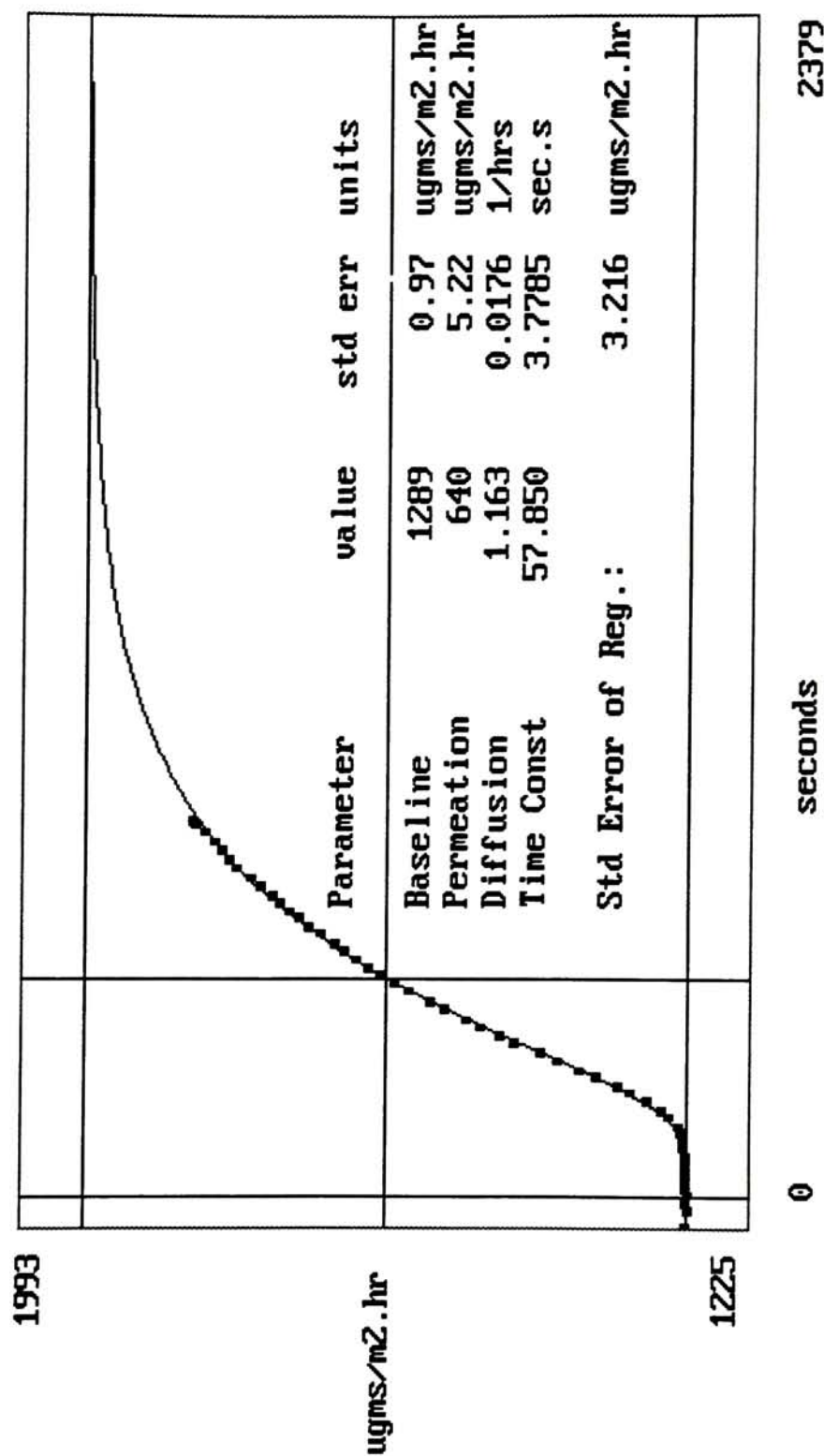


Figure 20

material: Met-HB
 caliper: .7
 permeant: linalool
 concentration: 1
 cell temp: 90
 fid temp: 150

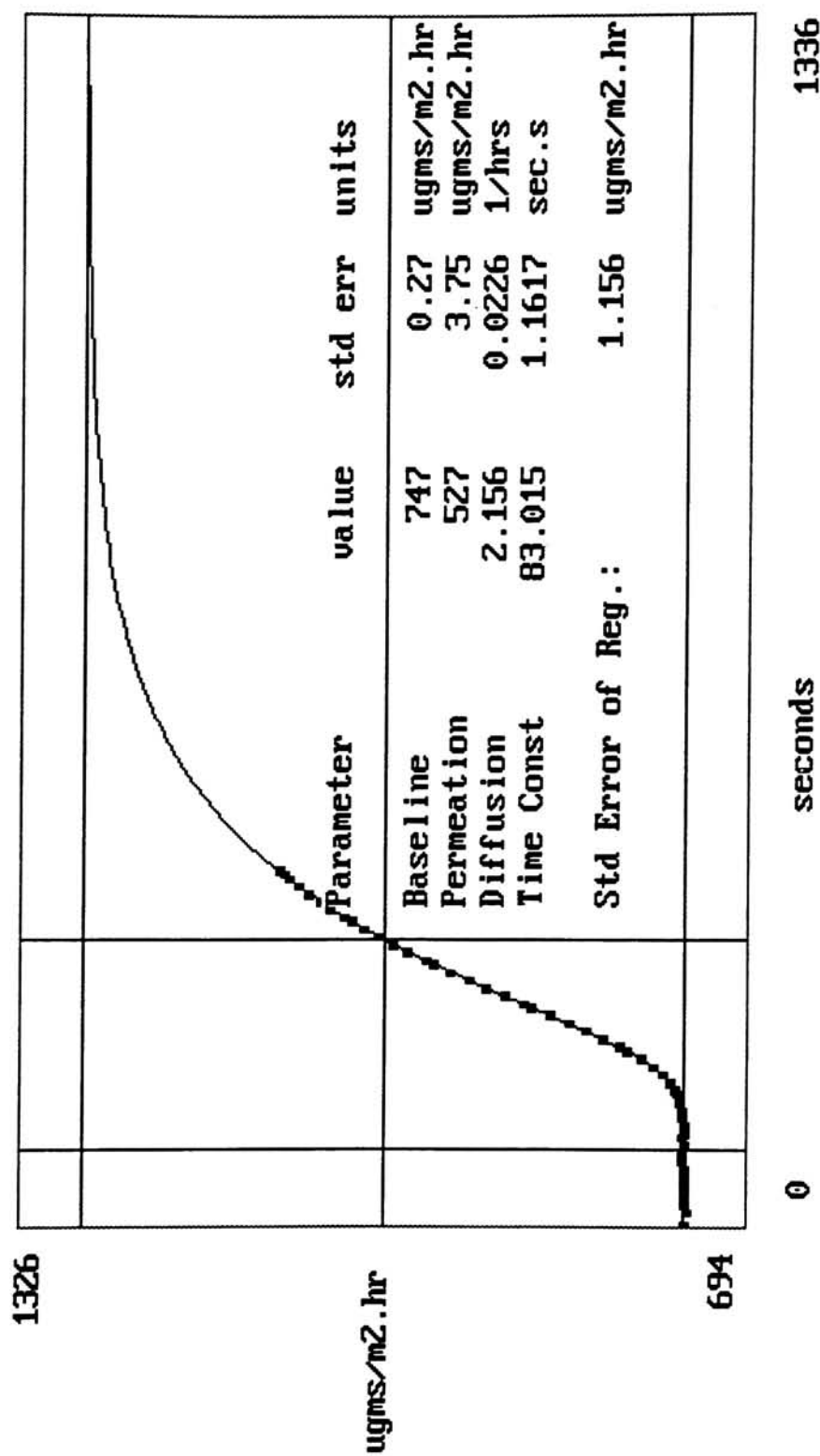


Figure 21

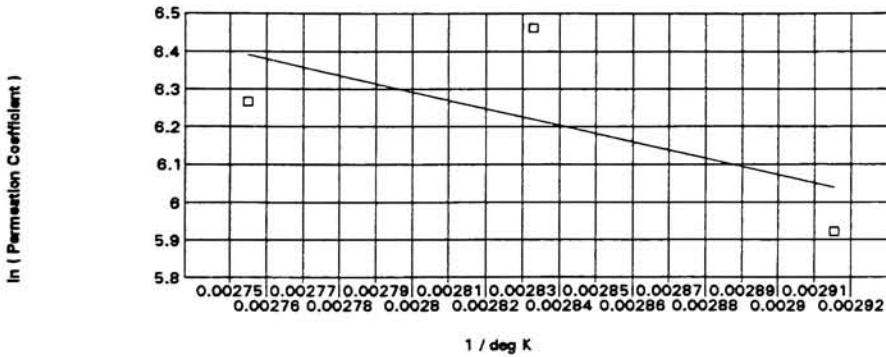
MAS2000 Temperature Analysis MAS Technologies

Material: **Met-HB**
Caliper (mils): **0.7**

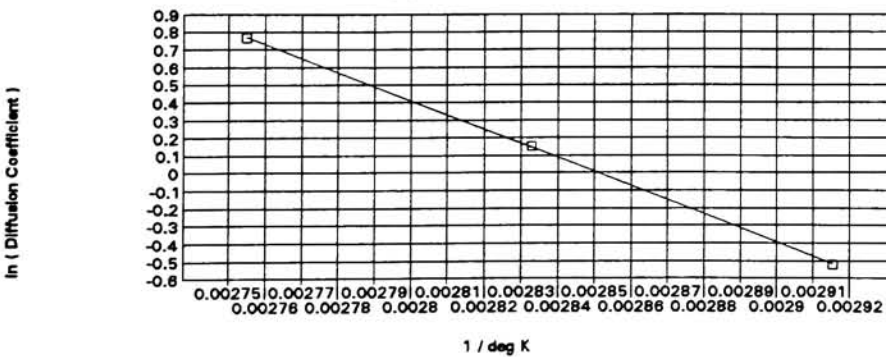
Permeant: **linalool**
Concentration: **1.00**

Permeation Units: **ugms/m2.hr**
Diffusion Units: **1/hrs**
Solubility Units: **ugms/m2**

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

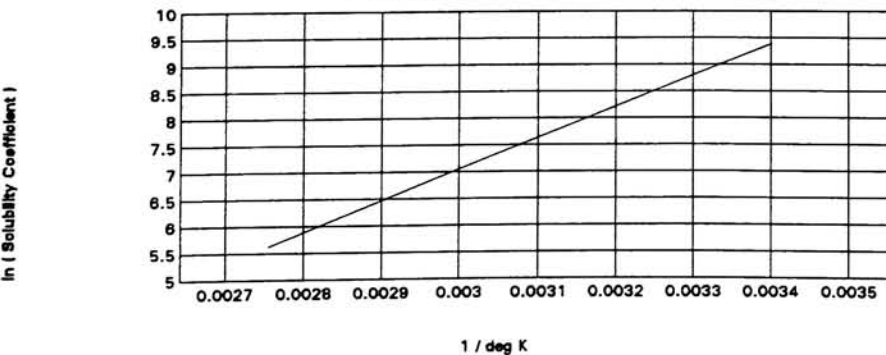


Figure 22

material: control
caliper: 1

permeant: linalool
concentration: 1

cell temp: 70
fid temp: 150

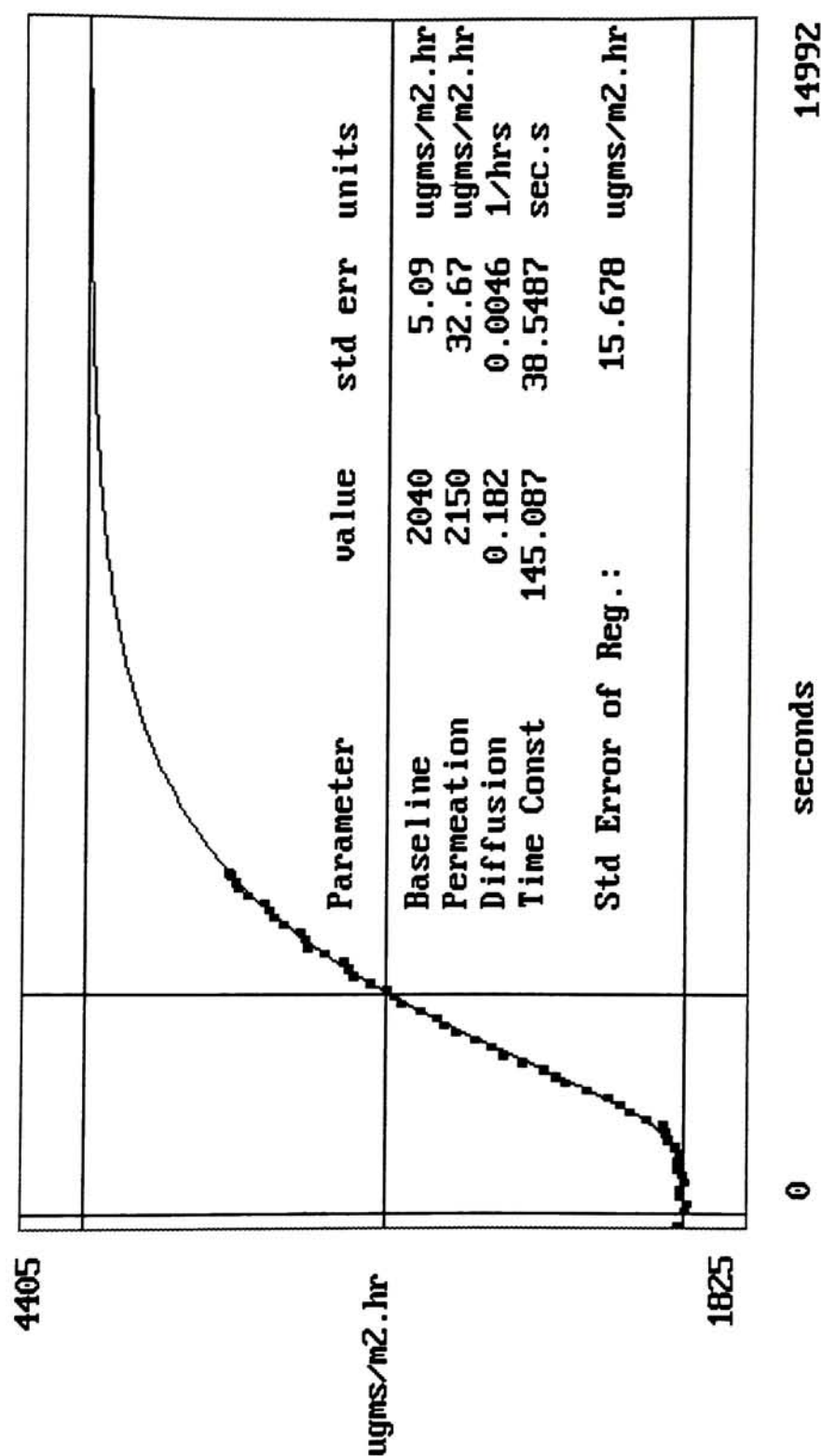


Figure 23

material: control
caliper: 1

permeant: linalool
concentration: 1

cell temp: 80
fid temp: 150

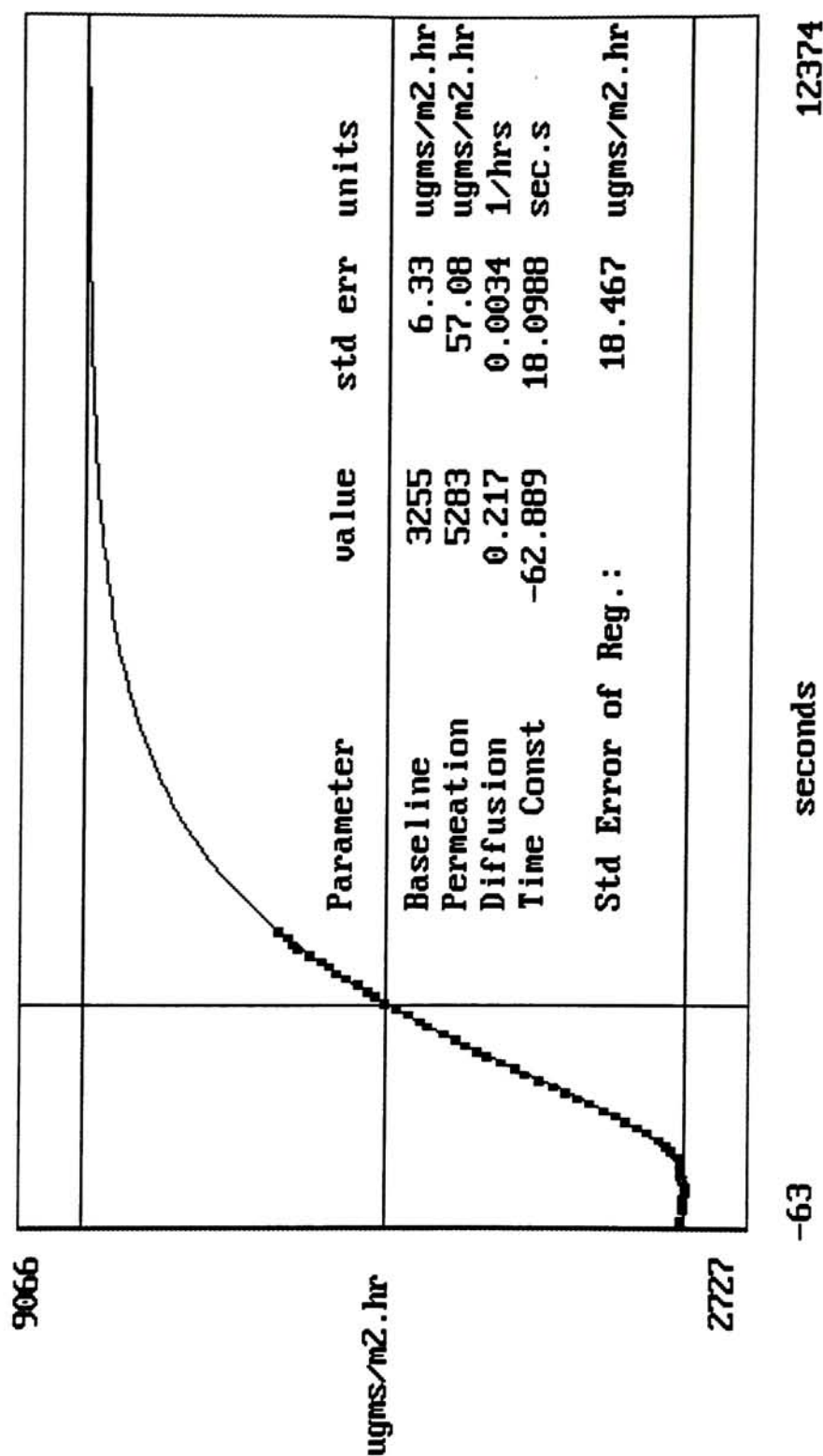


Figure 24

material: control
caliper: 1

permeant: linalool
concentration: 1
cell temp: 90
fid temp: 150

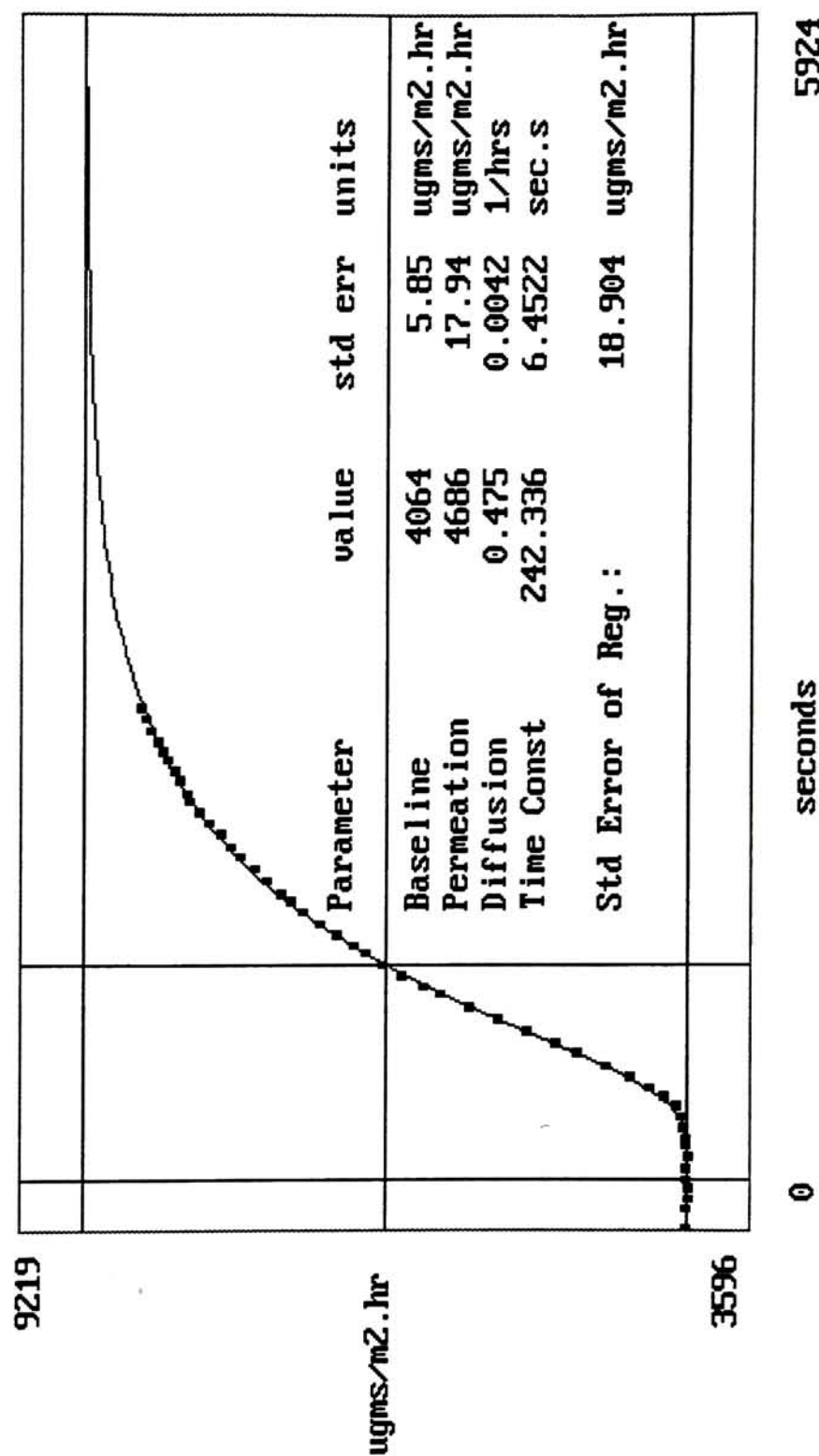


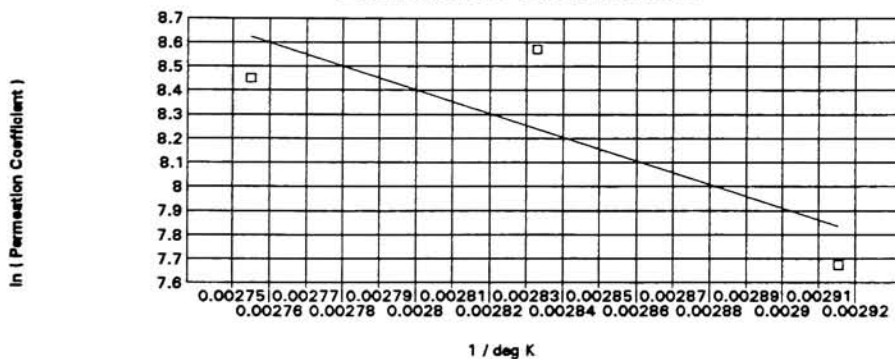
Figure 25

Material: *control*
Caliper (mils): *1*

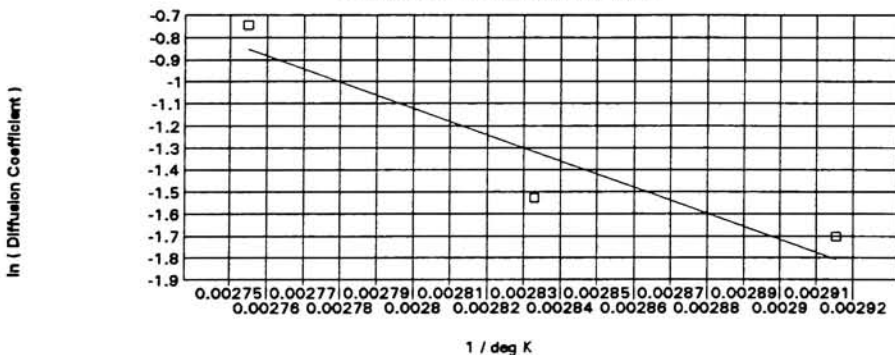
Permeant: *linalool*
Concentration: *1.00*

Permeation Units: *ugms/m2.hr*
Diffusion Units: *1/hrs*
Solubility Units: *ugms/m2*

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

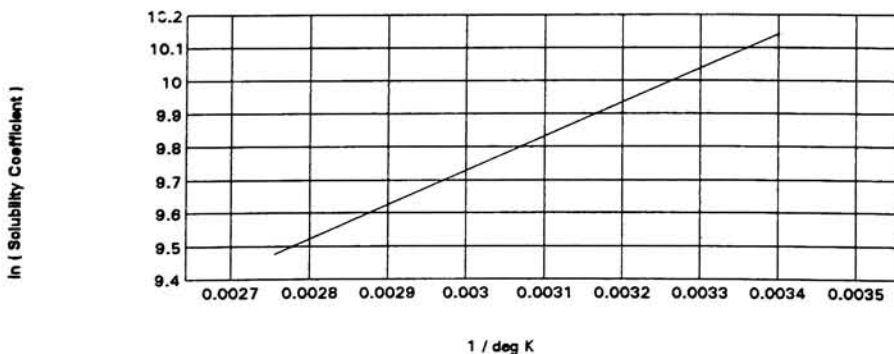


Figure 26

File: BSR0112A

Calibration File: ABX0110A

material: BSR
caliper: .7

permeant: linalool
concentration: 1
cell temp: 60
fid temp: 150

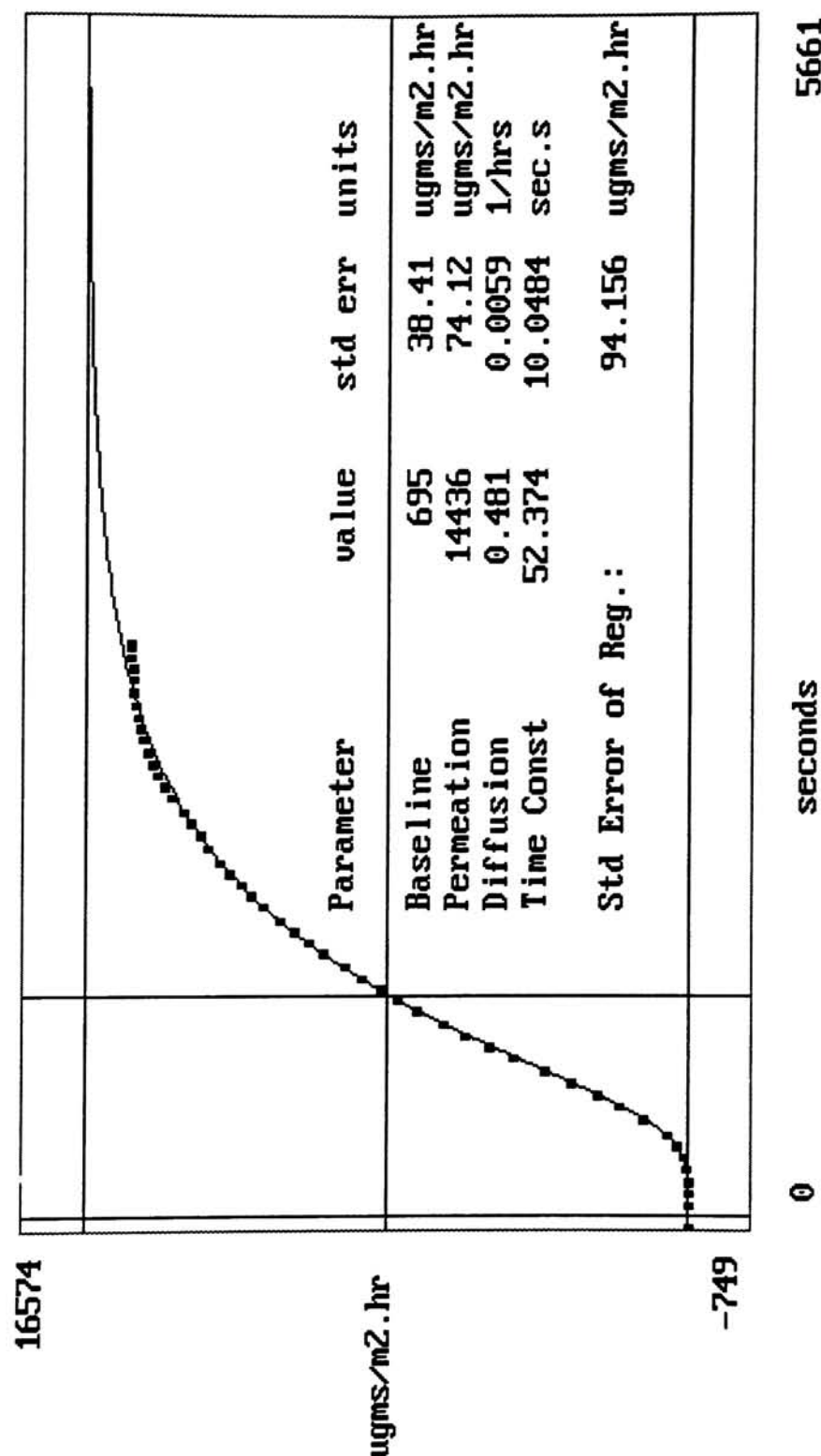


Figure 27

f1:save f2:report setup f3:exit

material: BSR
caliper: .7

permeant: linalool
concentration: 1

cell temp: 70
fid temp: 150

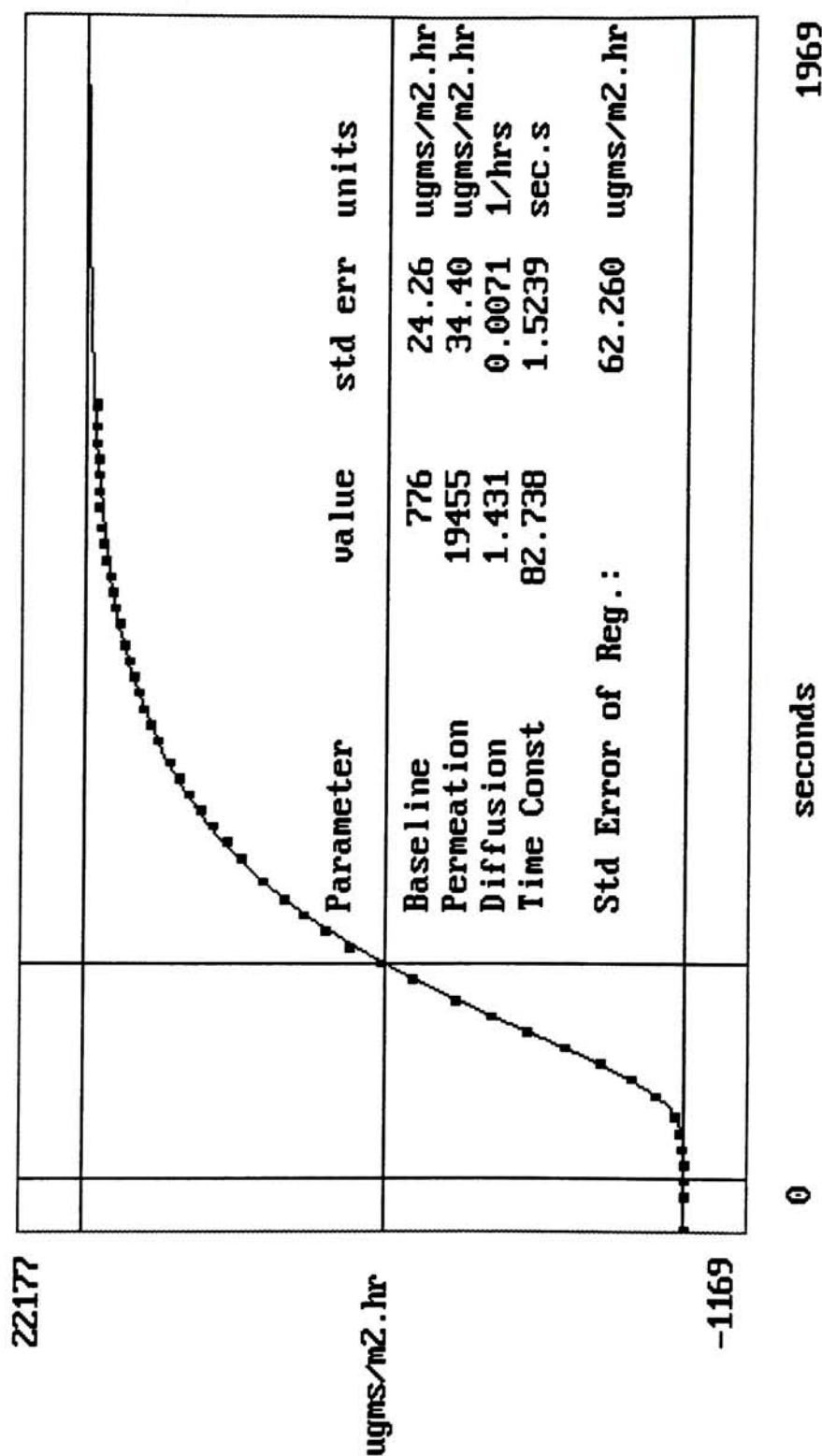


Figure 28

File: BSR0111A

Calibration File: ABX0110A

material: 3SR
caliper: .7

permeant: linalool
concentration: 1
cell temp: 80
fid temp: 150

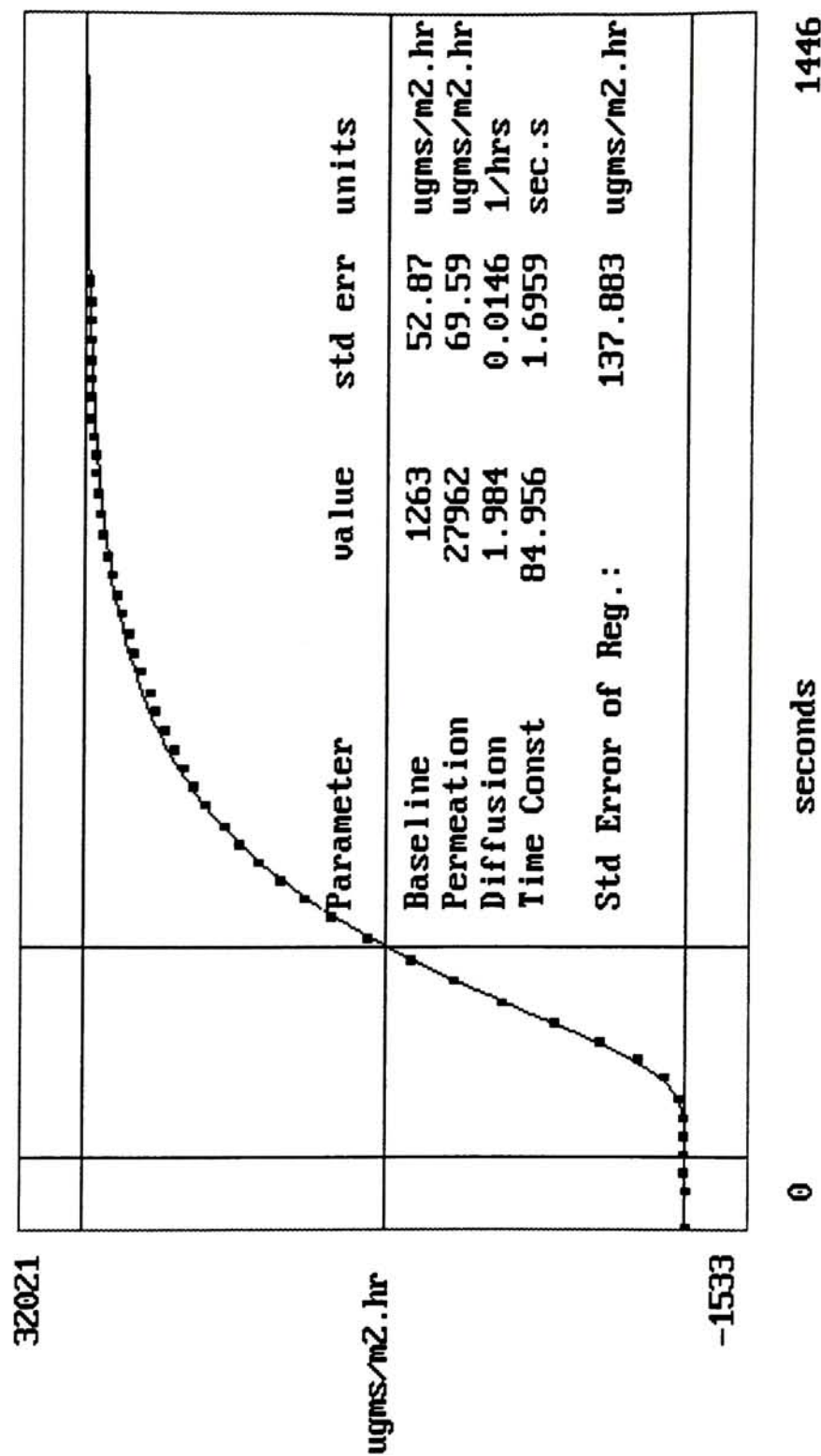


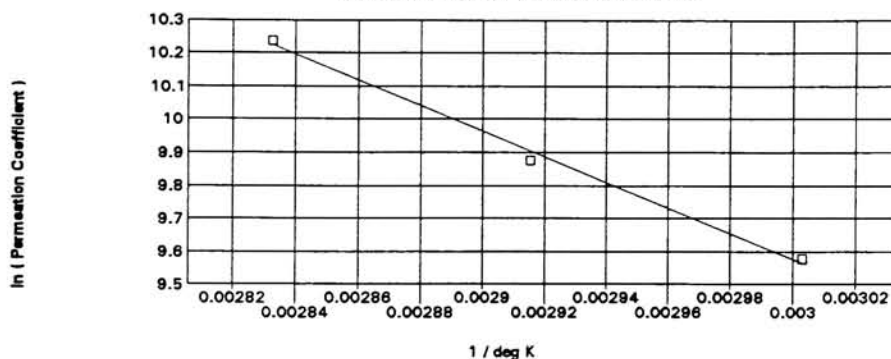
Figure 29

Material: **BSR**
Caliper (mils): **0.7**

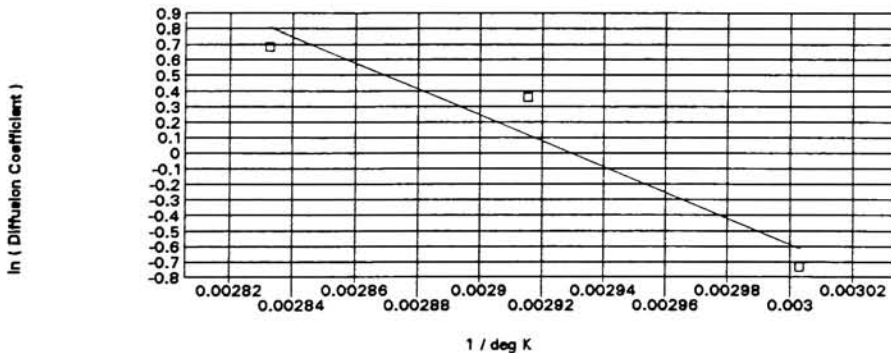
Permeant: **linalool**
Concentration: **1.00**

Permeation Units: **ugms/m2.hr**
Diffusion Units: **1/hrs**
Solubility Units: **ugms/m2**

Permeation Coefficients



Diffusion Coefficients



Solubility Coefficient Estimate

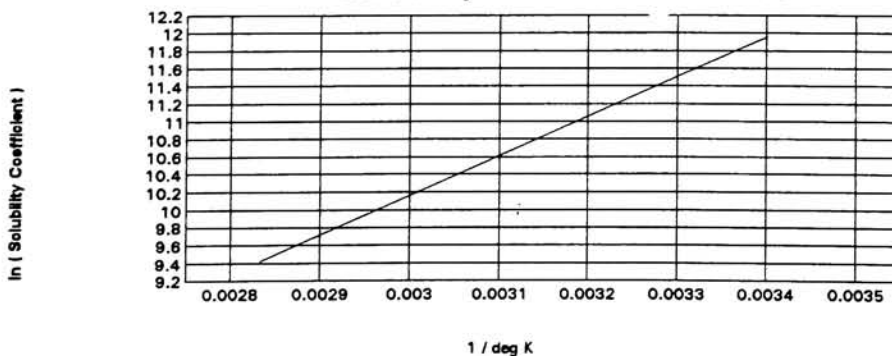


Figure 30

Material:	<i>ABX</i>
Caliper (mils):	<i>0.75</i>

Permeant:	<i>linalool</i>
Concentration:	<i>1.00</i>

Permeation Units:	<i>ugms/m2.hr</i>
Diffusion Units	<i>1/hrs</i>
Solubility Units:	<i>ugms/m2</i>

Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

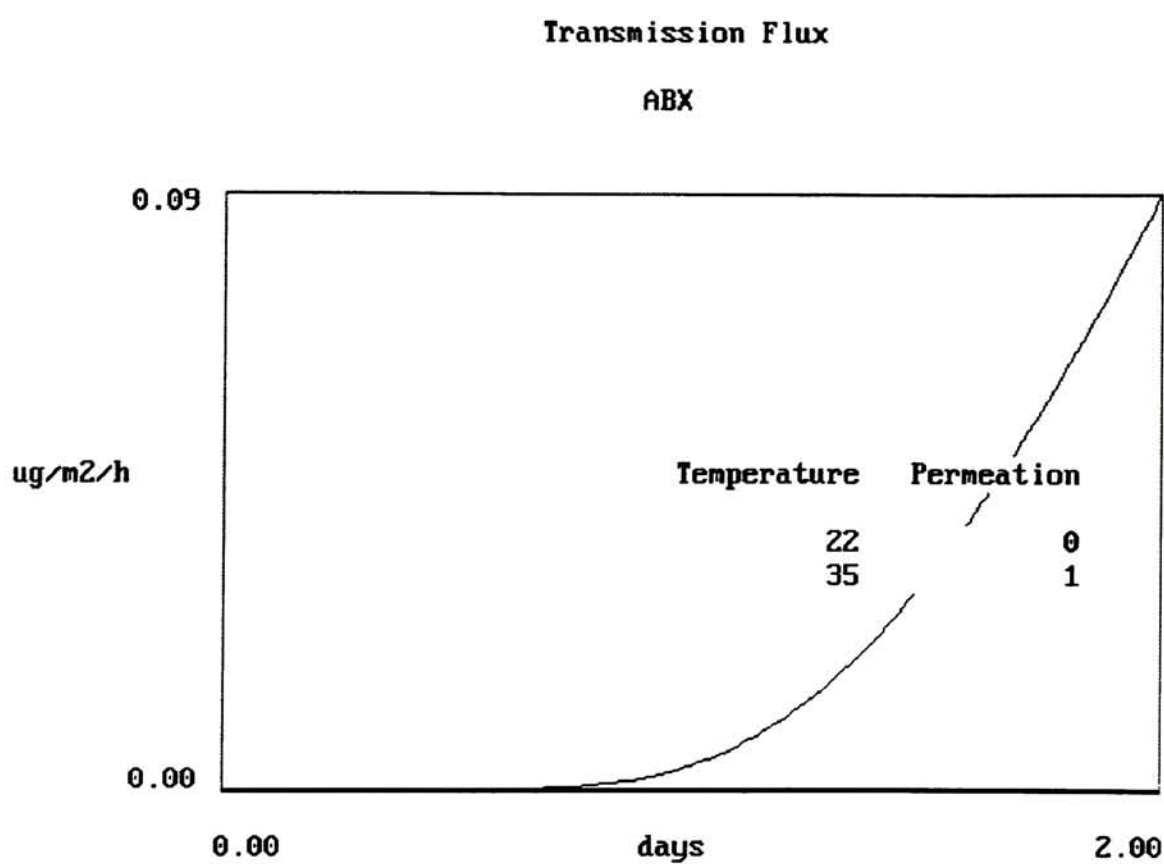
Intercept Slope

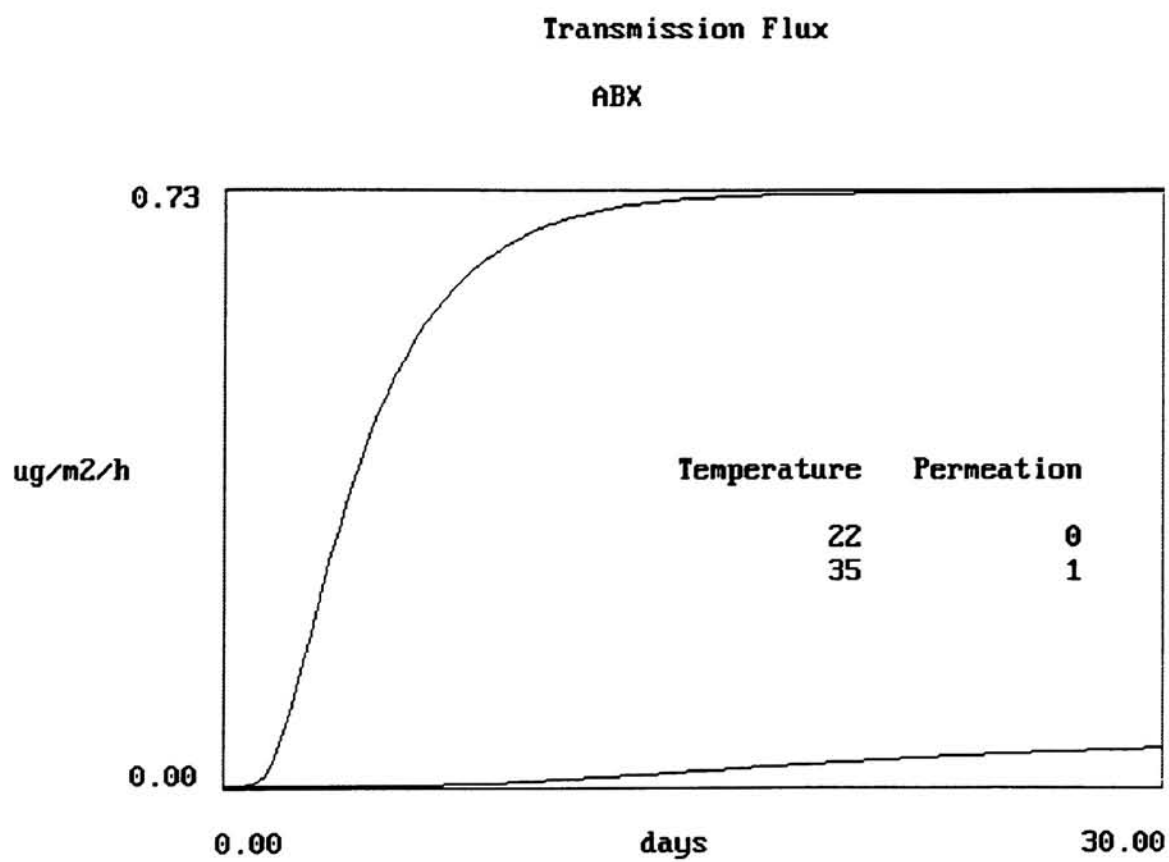
Permeation	<i>53.73225</i>	<i>-16644.5</i>
Diffusion	<i>32.60112</i>	<i>-12052.4</i>

Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	<i>0.734454 ugms/m2.hr</i>
Diffusion	<i>0.001458 1/hrs</i>
Solubility	<i>503.5004 ugms/m2</i>

Figure 31

**Figure 32**

**Figure 33**

Material:	<i>HBS</i>
Caliper (mils):	<i>0.7</i>

Permeant:	<i>linalool</i>
Concentration:	<i>1.00</i>

Permeation Units:	<i>ugms/m2.hr</i>
Diffusion Units	<i>1/hrs</i>
Solubility Units:	<i>ugms/m2</i>

Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

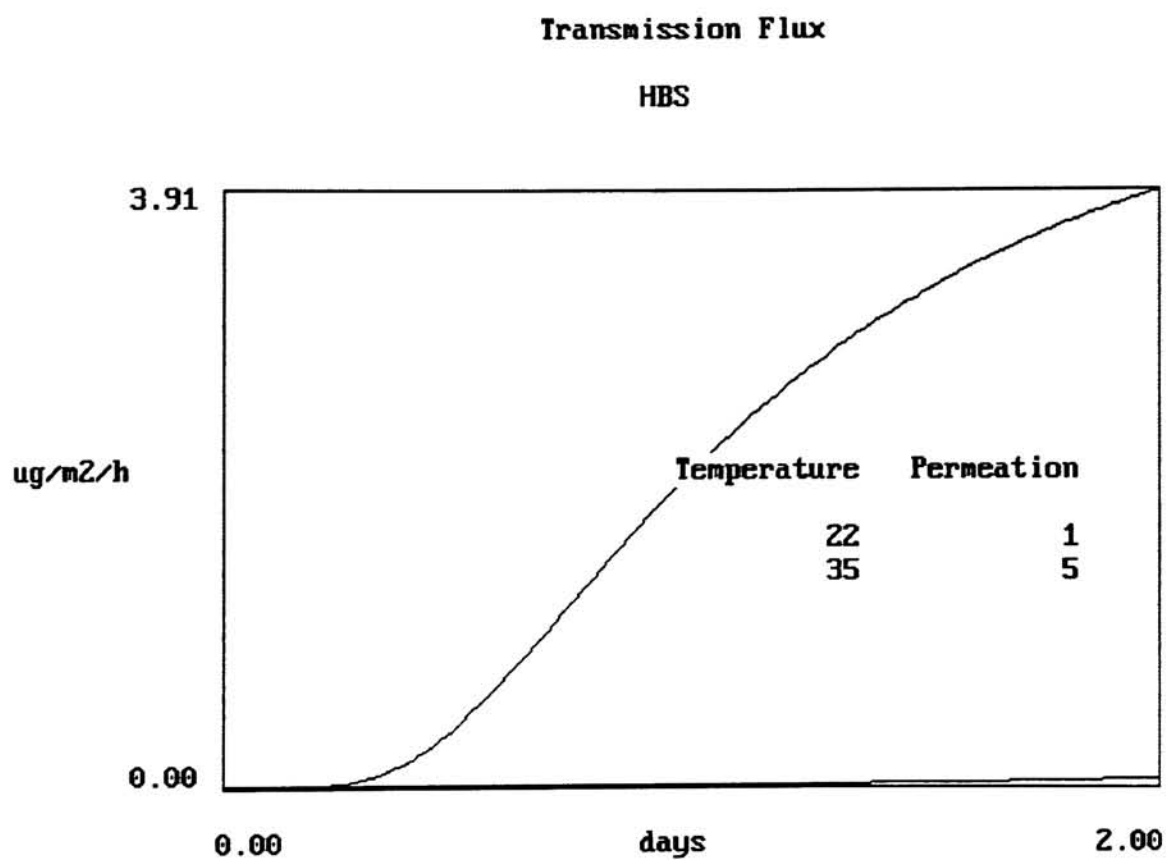
Intercept Slope

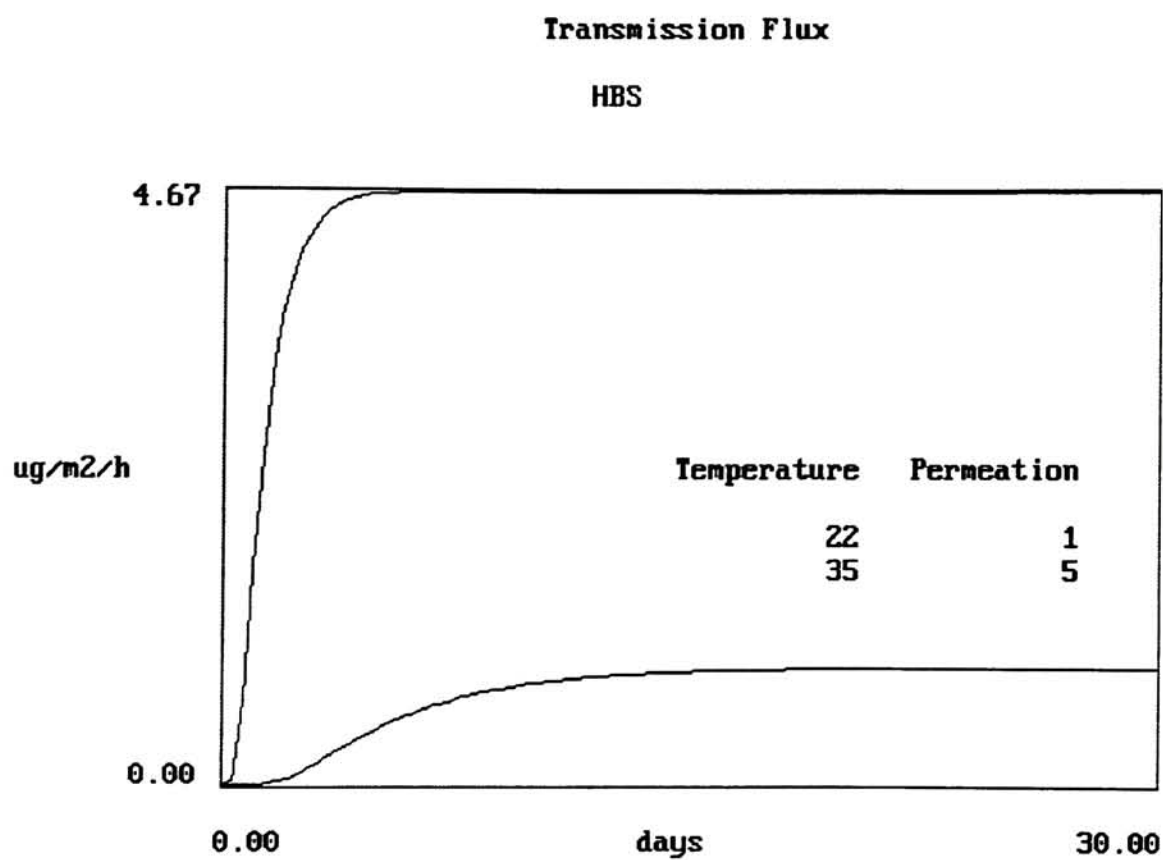
Permeation	<i>38.32158</i>	<i>-11328.6</i>
Diffusion	<i>29.78614</i>	<i>-10788.0</i>

Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	<i>4.665751 ugms/m2.hr</i>
Diffusion	<i>0.005300 1/hrs</i>
Solubility	<i>880.2273 ugms/m2</i>

Figure 34

**Figure 35**

**Figure 36**

MAS2000 Temperature Analysis	MAS Technologies
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Material:	60 MAC
Caliper (mils):	0.6

Permeant:	linalool
Concentration:	1.00

Permeation Units:	ugms/m2.hr
Diffusion Units	1/hrs
Solubility Units:	ugms/m2

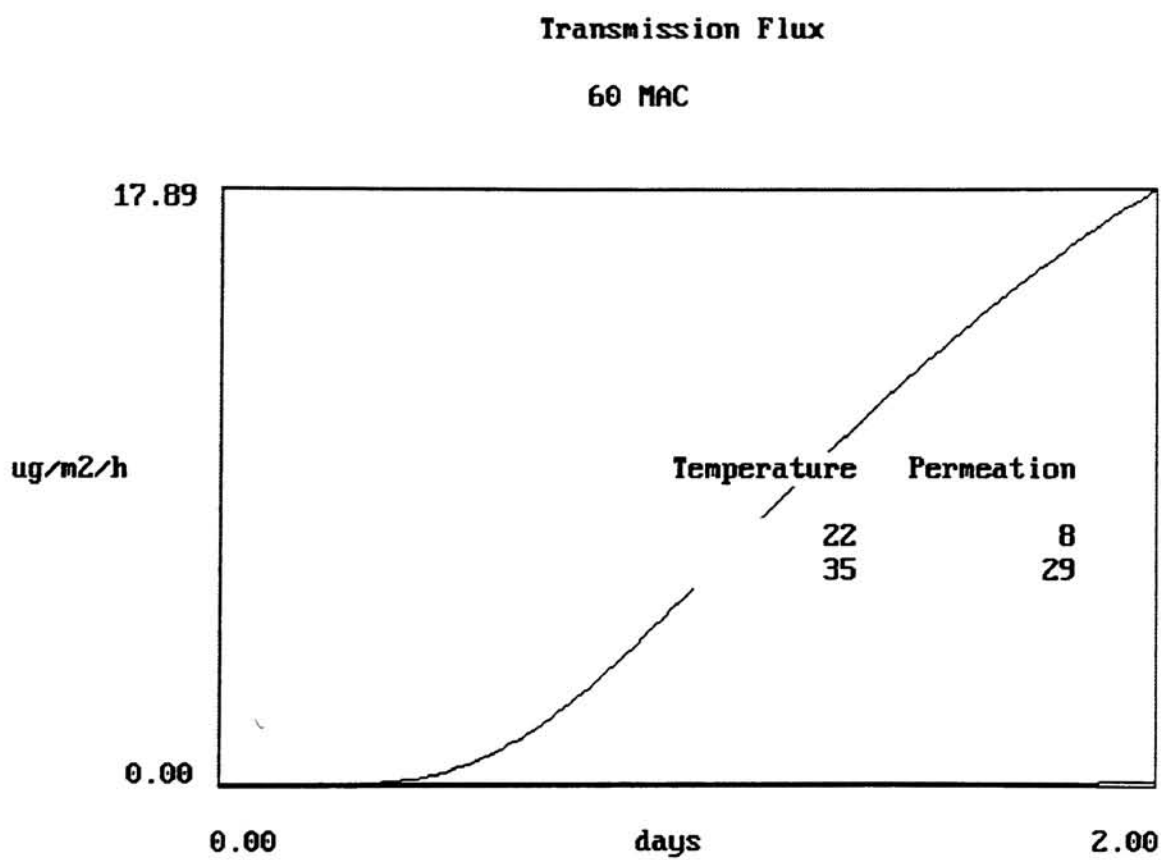
Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

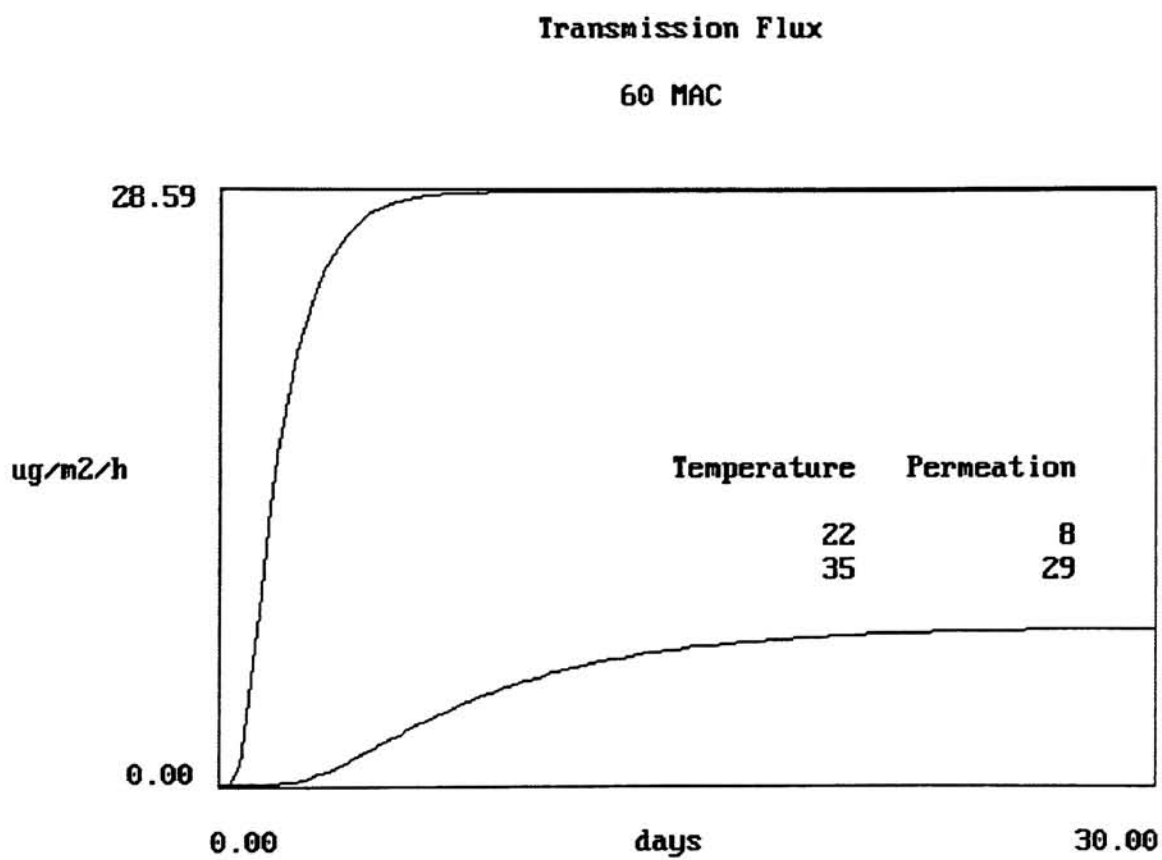
	Intercept	Slope
Permeation	33.58859	-9312.52
Diffusion	29.12812	-10711.1

Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	28.59150 ugms/m2.hr
Diffusion	0.003523 1/hrs
Solubility	8113.528 ugms/m2

Figure 37

**Figure 38**

**Figure 39**

Material:	<i>Met-HB</i>
Caliper (mils):	<i>0.7</i>

Permeant:	<i>linalool</i>
Concentration:	<i>1.00</i>

Permeation Units:	<i>ugms/m2.hr</i>
Diffusion Units:	<i>1/hrs</i>
Solubility Units:	<i>ugms/m2</i>

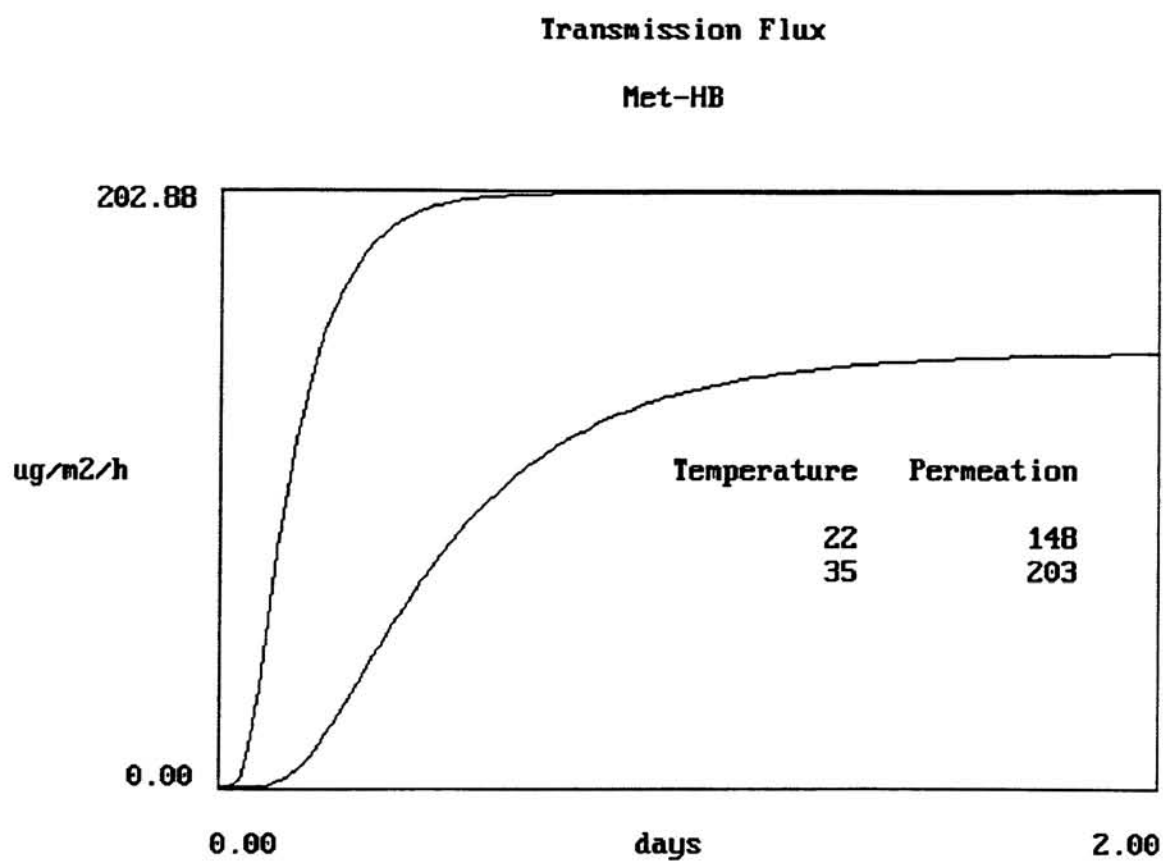
Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

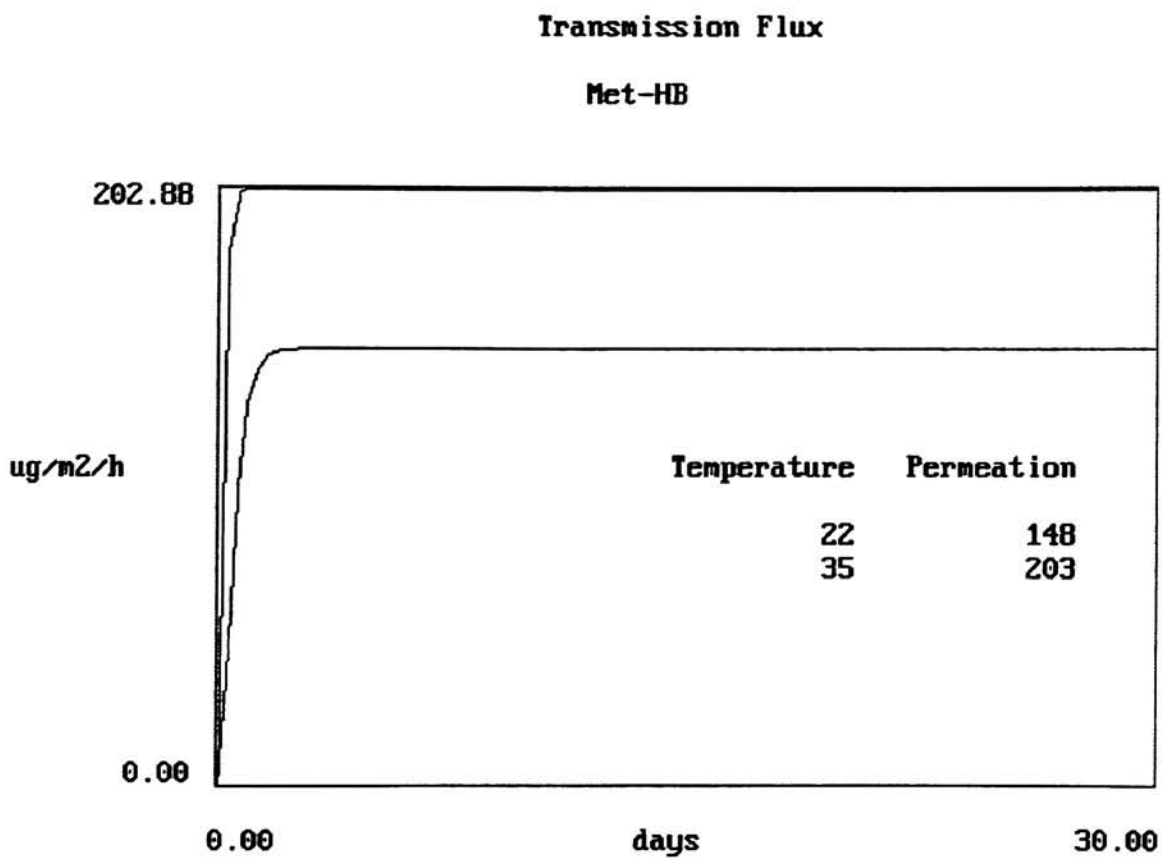
	Intercept	Slope
Permeation	<i>12.43097</i>	<i>-2192.45</i>
Diffusion	<i>22.91908</i>	<i>-8039.54</i>

Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	<i>202.8776 ugms/m2.hr</i>
Diffusion	<i>0.041447 1/hrs</i>
Solubility	<i>4894.817 ugms/m2</i>

Figure 40

**Figure 41**

**Figure 42**

Material:	<i>control</i>
Caliper (mils):	<i>1</i>

Permeant:	<i>linalool</i>
Concentration:	<i>1.00</i>

Permeation Units:	<i>ugms/m2.hr</i>
Diffusion Units	<i>1/hrs</i>
Solubility Units:	<i>ugms/m2</i>

Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

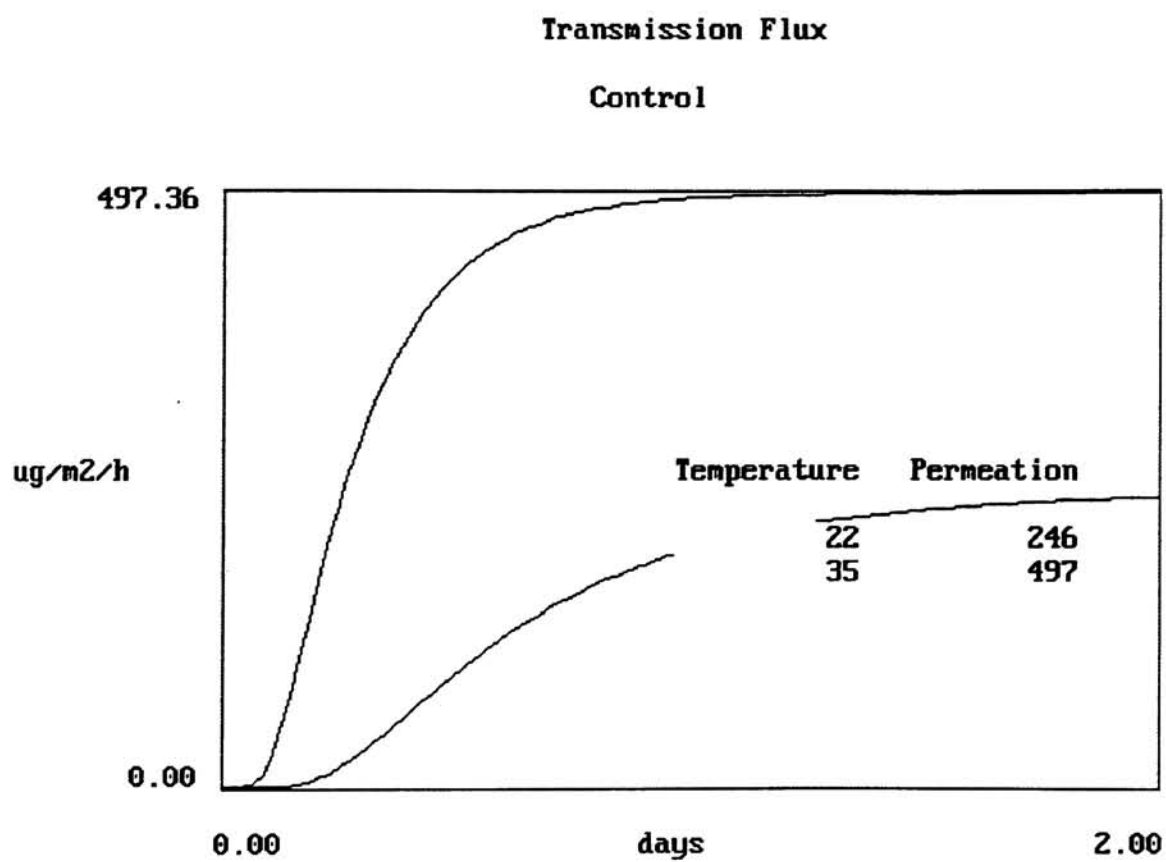
Intercept Slope

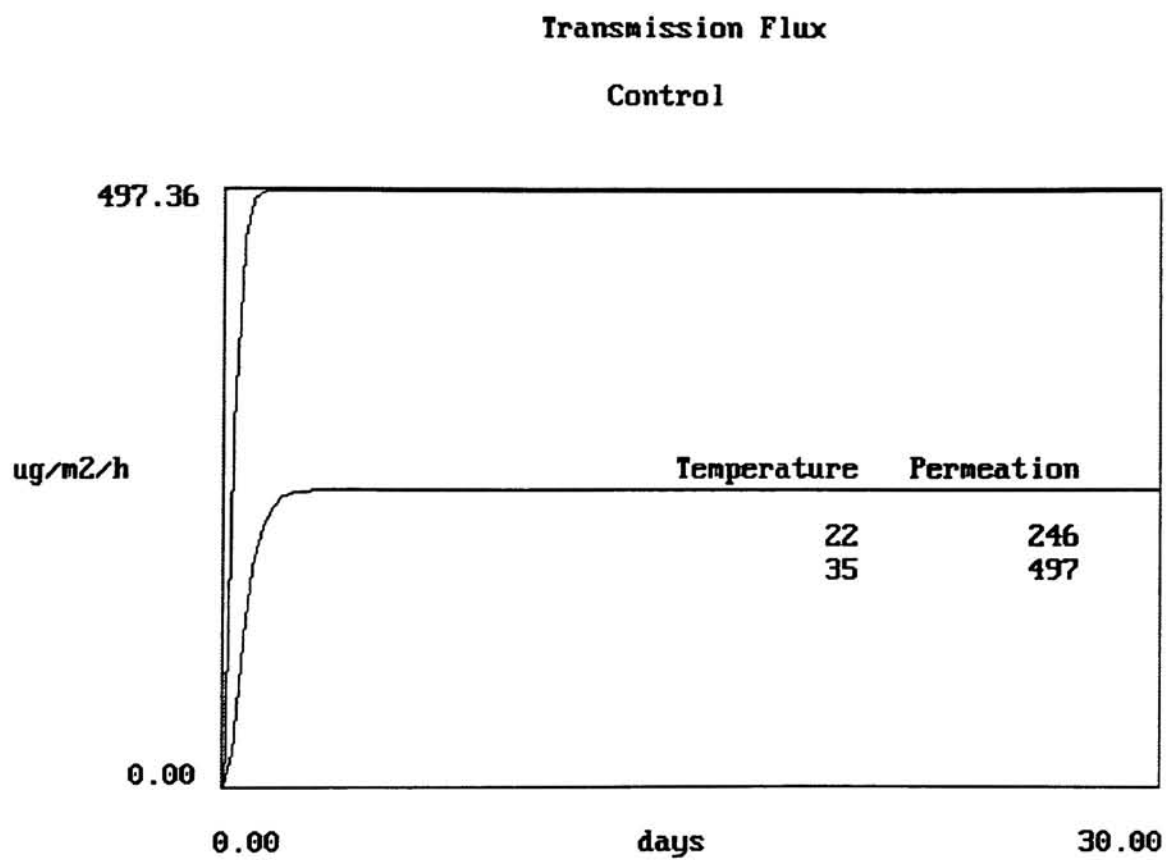
Permeation	<i>22.1395</i>	<i>-4906.50</i>
Diffusion	<i>15.51379</i>	<i>-5941.02</i>

Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	<i>497.3474 ugms/m2.hr</i>
Diffusion	<i>0.022931 1/hrs</i>
Solubility	<i>21688.36 ugms/m2</i>

Figure 43

**Figure 44**

**Figure 45**

Material:	BSR
Caliper (mils):	0.7

Permeant:	linalool
Concentration:	1.00

Permeation Units:	ugms/m2.hr
Diffusion Units:	1/hrs
Solubility Units:	ugms/m2

Arrhenius Fit
Regression Parameters
 $\ln(P) = \text{int.} + \text{slp.} / T$

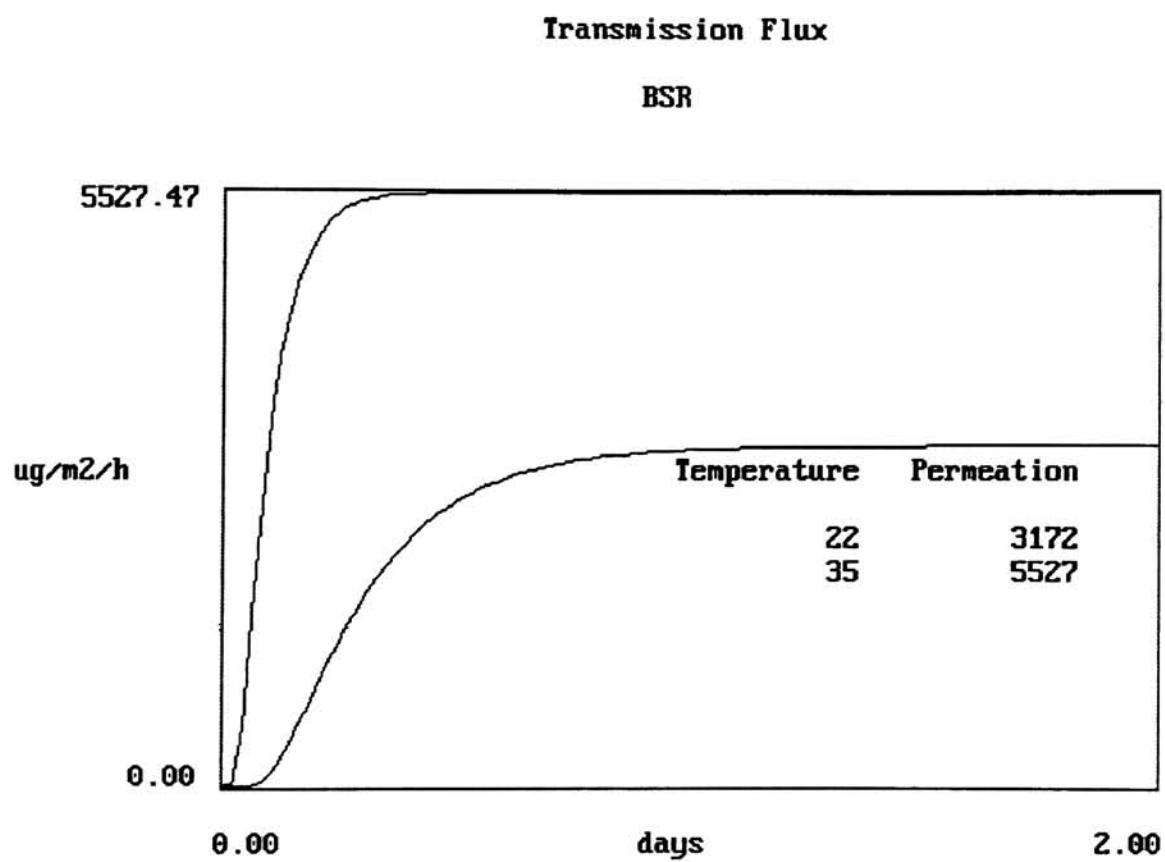
Intercept Slope

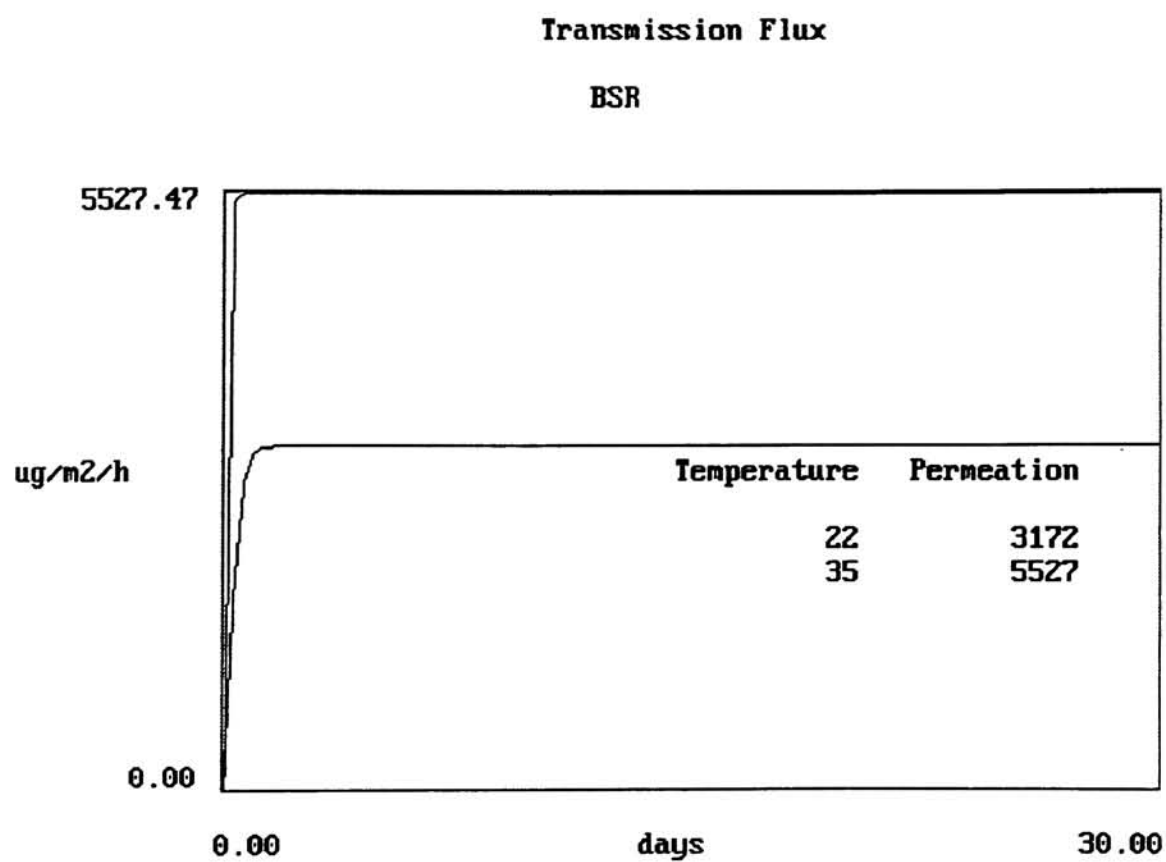
Permeation	21.22268	-3882.40
Diffusion	24.50715	-8365.51

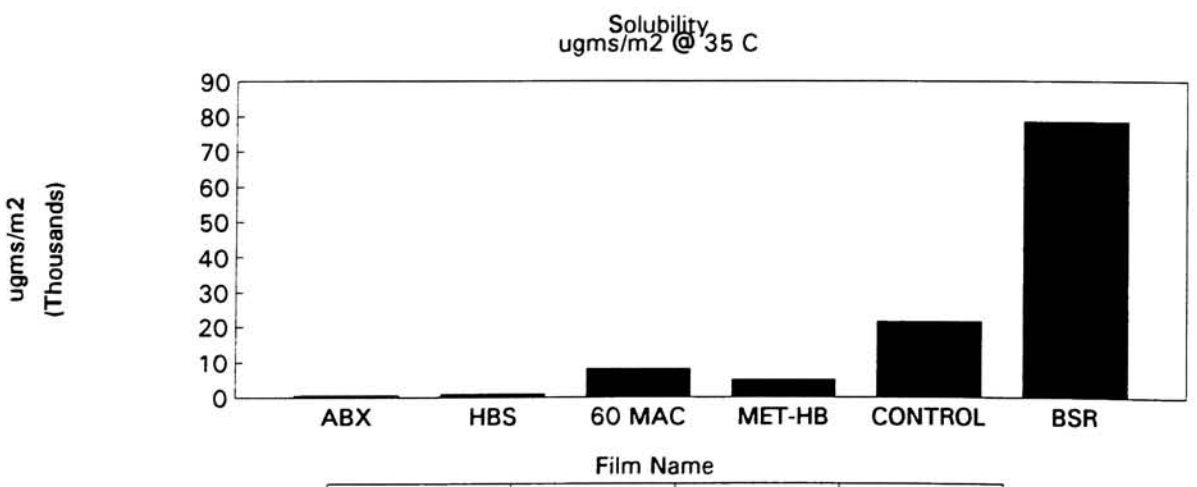
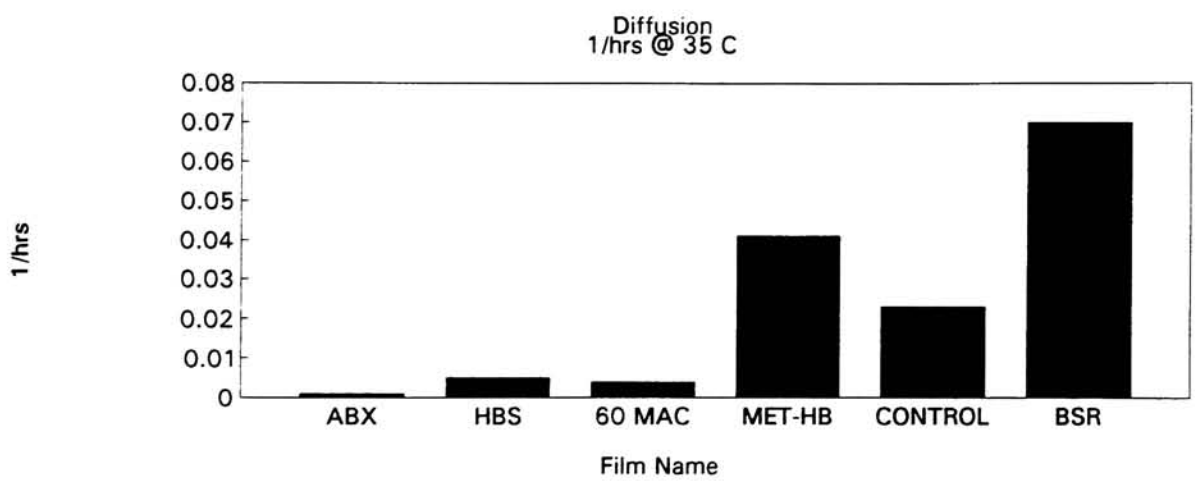
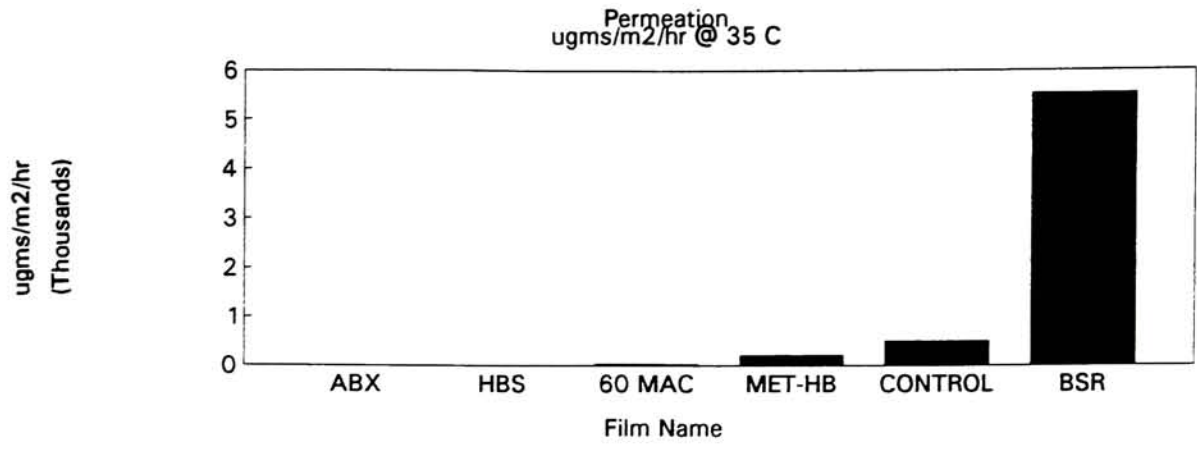
Arrhenius Fit
Room Temperature Projection (35 deg. C)

Permeation	5527.450 ugms/m2.hr
Diffusion	0.070397 1/hrs
Solubility	78517.90 ugms/m2

Figure 46

**Figure 47**

**Figure 48**



Film Name	Perm.	Diff.	Sol.
ABX	0.73	0.001	503
HBS	4.67	0.005	880
60 MAC	28.59	0.004	8113
MET-HB	202.88	0.041	4894
CONTROL	497.35	0.023	21688
BSR	5527.45	0.07	78517

Figure 49

Film Name	Time to Equilibrium (days)
ABX	14.29
HBS	3.93
60 MAC	5.91
MET-HB	0.5
CONTROL	0.91
BSR	0.3

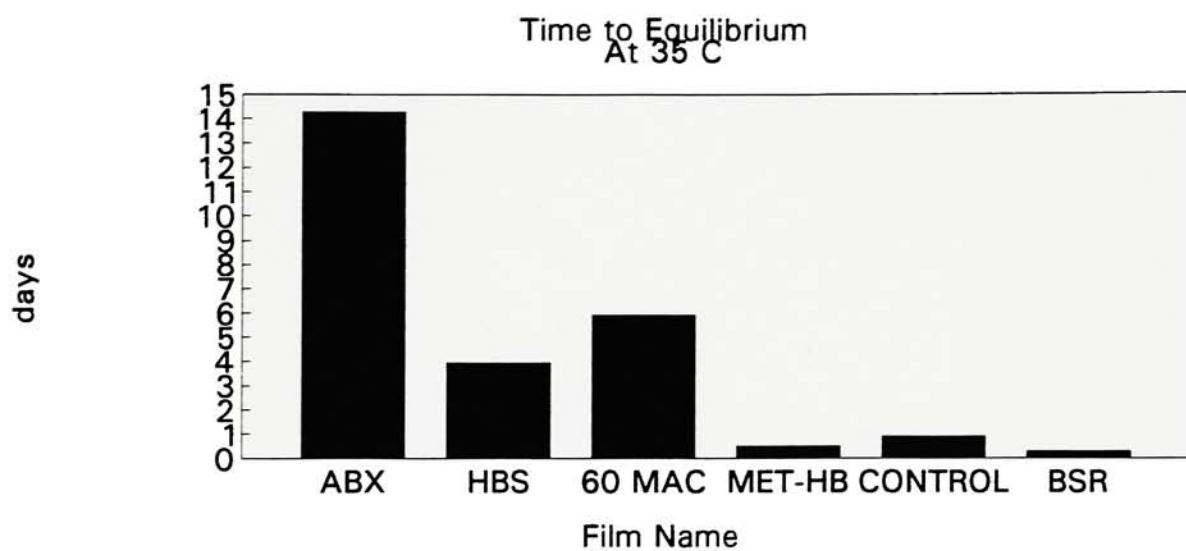


Figure 50